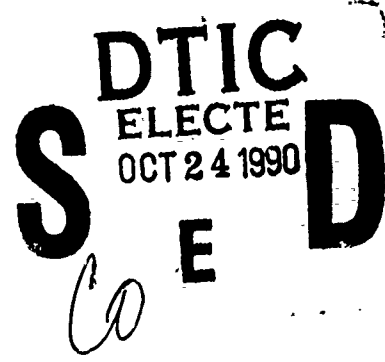


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# ACCURACY OF CASTING SINGLE CROWNS IN TITANIUM

A

THESIS

Presented to the Faculty of  
The University of Texas Graduate School of Biomedical Sciences  
at San Antonio  
in Partial Fulfillment  
of the Requirements  
for the Degree, of  
MASTER OF SCIENCE

by

CHRISTOPHER MELVIN MINKE, B.S., D.M.D.

San Antonio, Texas

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ACCURACY OF CASTING SINGLE CROWNS IN TITANIUM

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DEAN

## DEDICATION

This thesis is dedicated to my family. My wife, Marilyn, and my two sons, Andrew and Todd, sacrificed and supported me totally in my effort to complete this work. Their understanding permitted me the necessary time and gave the encouragement I needed throughout my residency.

### ACKNOWLEDGMENTS

I would like to acknowledge Dr. Ronald B. Blackman (Associate Clinical Professor, Dept. of Restorative Dentistry) for his interest in this research project. His enthusiasm and instruction in casting titanium provided me with the impetus and ability to complete this phase of my residency.

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ACCURACY OF CASTING

SINGLE CROWNS

IN TITANIUM

Christopher M. Minke, M.S.

The University of Texas Graduate School of Biomedical Sciences  
at San Antonio

Supervising Professor: E. Steven Duke. D.D.S., M.S.D.

✓  
For an alternative metal to be useful in dentistry it should be strong, biocompatible, low cost, and easy to handle. It must also be possible to cast the metal accurately and predictably. The purpose of this investigation was to determine the casting accuracy of 99.5% commercially pure titanium for dental crowns.

Single crowns were cast in the configuration of a metal ceramic restoration for a maxillary left cuspid. The master pattern was waxed to full contour and cut back to represent the standard clinical metal ceramic restoration. Crowns were cast in titanium, nickel-chromium alloy, and gold-palladium alloy. The castings were then luted to the dies on which they were waxed, embedded in clear resin, and sectioned.

(15) ✓

The linear width of the cement space at specific locations around the sectioned crown and die assemblies was determined with a measuring microscope. A comparison of the mean cement space of titanium was made with the mean cement space of both gold-palladium alloy and nickel-chromium alloy.

The mean cement space for titanium fell between that of the gold-palladium and nickel-chromium alloys. Statistically, there was no significant difference in the casting accuracy of gold-palladium alloy and titanium. Both gold-palladium and titanium, however, were significantly more accurate than the nickel-chromium alloy in the multiple range test at the 0.050 level.

Based on this investigation, commercially pure titanium can be cast as accurately as gold-palladium alloy. It meets the criteria of low cost, favorable physical properties, and biocompatibility. The casting process used in this study, however, was found to be technique sensitive.



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## I. INTRODUCTION

A major weakness of cast crown restorations can be the adaptation of the casting to the prepared tooth. The closeness with which the crown fits will affect the seating of the crown, the subsequent cement space, the adaptation at the margins, and the occlusion (Windler, 1979). Under ideal circumstances, a casting exactly reproducing the wax pattern in every dimension is necessary for precise fit and occlusion. Therefore, alloys for cast dental restorations must be accurate when cast by the "lost wax" process.

William H. Taggart (1907) revolutionized dental casting with a unique process and claimed his crowns fit every time regardless of the metal used. In the eighty-three years that have followed Taggart's invention, a "perfect fit" has remained elusive.

High noble alloys have been the best performers over the years for degree of fit, durability, and biocompatibility. If the cost of gold were still \$35.00 per ounce, base metal alloys would not engage the attention of dental researchers to the extent they have today (Naylor, 1986). The quest for a metal that will achieve the same success as gold at a lower cost is unfulfilled.

Titanium is a metal familiar to the aerospace industry because of its high strength to weight ratio and low cost. Interest in the use of titanium in dentistry increased as a result of the work by P.I Branemark, who discovered

osseointegration during vital microscopic studies of microcirculation in bone (Branemark, et.al. 1985). Titanium's biocompatibility in dental implantology created an interest in its use in cast restorations.

The aerospace industry discovered the machining of titanium from wrought or forged stock to be a considerable waste of material. Techniques for "near net shape" processing were researched and developed to reduce the "buy-to-fly" ratio for titanium components (Whitt and Weaver, 1984). Casting titanium has provided the aerospace industry with a considerable cost savings, and dentistry with a possible alternative metal to high noble alloys.

Cast commercially pure titanium may have several advantages as an alternative to high noble alloys. With worldwide concerns over the potential health hazard of nickel, beryllium, chromium, cobalt, and copper, this base metal has proven biocompatibility. An oxide ceramic forms on the surface of titanium immediately upon contact with air. This surface oxide causes the metal to "passivate" in the oral environment minimizing further corrosion. Titanium's mechanical properties in the cast state are similar to type IV gold. It can be polished to a high luster and is burnishable. It has the advantage over gold of being considerably less expensive and has a specific gravity of approximately one-third that of gold (Ohara, 1987). This means that it is not only less expensive, but one ounce of titanium will yield three times as many castings as one ounce of gold.

The most significant obstacle to titanium's use as an alternative metal in dentistry is the difficulty with which it is cast. The pure metal melts at 1720 °C and is highly reactive with oxygen and nitrogen at this temperature. It is not only difficult to melt, but it has a casting shrinkage of approximately 2.5% (Leisner, 1984). The shrinkage from solidification to room temperature for noble dental alloys ranges from 1.25 - 1.6% , and for base metal dental alloys as much as 2.0% (Craig, 1985). Therefore, the compensating expansion of standard dental investments is inadequate and the investment / metal interaction at the high casting temperature is considerable. Currently, casting parameters account for the majority of dental research involving titanium.

This research project was undertaken to determine accuracy of casting single crowns in commercially pure (99.5%) titanium. The fit of castings on the dies on which the wax patterns were developed was measured and comparisons made with a high noble alloy and a base metal alloy. Since titanium's mechanical properties and biocompatibility are extremely favorable, it would serve as an excellent low cost alternative to the currently available noble metal alloys if it can be cast accurately and predictably.



Research Hypothesis

The casting accuracy of commercially pure titanium crowns is significantly different from the casting accuracy of gold-palladium and/or nickel-chromium crowns.  $P < 0.050$ .

Null Hypothesis

There is no difference in the casting accuracy of single crowns cast in commercially pure titanium, gold-palladium alloy, or nickel-chromium alloy.  $P < 0.050$ .

## II. LITERATURE REVIEW

### A. History of Dental Casting

The use of titanium in dentistry is in its infancy. By examining the history of cast dental restorations it is possible to understand the "growing pains" titanium is experiencing as an emerging alternative metal for dental restorations.

On January 15, 1907, Dr. William H. Taggart presented his casting machine and technique to the New York Odontological Society. He boasted a "perfect fit" regardless of the metal used (Taggart, 1907). Taggart's presentation initiated great interest in the casting process for dental restorations and the development of casting machines. Prior to 1907, gold crowns were commonly fabricated by soldering gold bands to occlusal surfaces that had been formed by swagging gold into metal die plates. This "gold shell" technique was first introduced by Morrison in 1869 (Malone, 1978).

Although Taggart popularized the casting process for dental restorations, the casting technique was certainly not new. There is evidence of bronze statues being cast by the Babylonians in 2230 B.C. using the "lost wax" or "wasting wax" technique. In Greece, around the eighteenth century B.C., Lycippus was known for his works in bronze using the "cire perdue" or "disappearing wax model" process (Stryker, 1911). The application of casting

techniques to the fabrication of dental restorations lead Stryker (1911) to conclude that dentistry "did nothing more than to revive an art that has been practiced in all branches of metal work from time immemorial, that probably is as old as man's knowledge of metals, and for which, at this late day, great claims for originality are made."

Gravity was the first force employed to make dental castings. Tin denture bases were cast by pouring molten metal into a mold by Dr. Edward Hudson of Philadelphia in 1820 (Hagman, 1976). In 1906, Dr. Solbrig of Paris introduced a mechanical casting device utilizing steam pressure. Dr. Solbrig based his device on the steam pressure technique of a Mr. Biber of Pforzheim, Germany (Platschick, 1908). Dr. William H. Taggart's nitrous oxide pressure casting machine employed a nitrous oxide flame to melt the metal along with gas pressure to force the molten metal into the mold (Hagman, 1976). His machine revolutionized prosthodontic casting in 1907.

Centrifugal force casting machines were developed by Campbell in 1908 using a cowbell and a chain (Campbell, 1911) and marketed in 1920 by Dental Products Company of Chicago. Also in the early 1900's, the first horizontal, spring loaded, centrifugal force casting machine was developed by Jameson and a vacuum casting machine call the "Elgin Pig" was developed by Ransom and Randolph (Hagman, 1976).

The investments of the early 1900's were primarily gypsum products and wax elimination was accomplished using boiling

water, steam, or torch heat. Sophisticated techniques of melting metal such as induction furnaces, helium arcs, and laser beams were developed in the 1930's (Hagman, 1976).

As cast dental restorations increased in popularity, problems with the fit of restorations became evident. Price(1909) noted that "pure gold " will contract 2.25% of its diameter in all directions. Price was the first to publish research data on the casting shrinkage of gold and waxes. He found that gold contracted 1.64% in transition from liquid to solid and 2.20% from solidification to room temperature and was the first to recognize that there must be compensation for this shrinkage (Price, 1911). Lane (1909) recommended pressure compensation (15 to 30 psi) to completely fill the mold and worked with different investment formulas to compensate for shrinkage. Van Horn (1910) was the first to advocate wax expansion to compensate for gold shrinkage and coined the term "wax pattern". As progress was slowly being made in the pursuit of accurate castings, Dr. Rhein of New York spoke of the average inlay as "a floating island of gold in a sea of cement."(Stryker, 1911).

In the 1920's, Volland (1928) advocated casting into a cold mold using low heat wax elimination at 320°F. Grunn (1928) used a long burnout with a heat soak, and cast when the center of the mold was cherry red. These articles appeared back-to-back in the same volume of the Journal of the American Dental Association indicating the controversy that raged on over casting techniques.

The first scientific evaluation of casting techniques was conducted by Taylor and Paffenbarger (1930). They evaluated seven different techniques: Volland (low heat elimination), Grunn (high heat elimination), Altfillish (rapid wax elimination by boiling), Van Horn (water bath technique), Meyer-Maves (mixing investment at 50°F above room temperature), Solbrig (multiple impressions of consecutive dies resulting in each subsequent die being larger than the preceding one), and Whip Mix (controlled water:powder ratio with thermal expansion). Castings were measured on steel dies designed by the Weinstein Laboratories for the National Bureau of Standards. Their conclusions were that thermal expansion was necessary and when the variables of investing and casting are correctly balanced, castings fit.

The variable affecting fit of cast dental restorations continued to be the focus of research. Taylor (1931) was the first to report on cristobalite investment developed by Sweeney and Paffenbarger. Scheu (1932) advocated hygroscopic expansion and found thicker mixes, investments mixed with hot water, and increased spatulation increased expansion up to 2.2%. The majority of the arguments over expansion techniques seemed to revolve around the thermal coefficient of expansion of gold. Values of 1.25% to as much as 3% were suggested by various researchers (Souder, 1934, Van Horn, 1934, Scheu, 1935). Phillips (1937) observed that the amount of expansion desired depended on whether the pattern was for an internal or external restoration.

Hollenback (1943) recommended a "hybrid investing and casting technique". He employed setting, hygroscopic, and thermal expansion with cristobalite investment and claimed consistent accuracy. Smyd (1955) recognized three methods of compensating for pattern and gold shrinkage: wax expansion, controlled thermal expansion, and hygroscopic expansion. Fusayama (1959) was the first to recognize a need to allow for cement film thickness and suggested a range of 5 to 30 microns. Skinner (1965) described the techniques of hygroscopic and thermal expansion, which are presently being used to consistently produce accurate castings with gold alloys.

#### B. Development of Alternative Alloys

The variables for producing accurate cast restorations with gold alloys are well understood and high gold content alloys are recognized as the profession's standard because of its ease of handling and biological acceptance in the oral environment (Eden, 1979, Byrne, 1986). In the 1950's, Dr. Abraham Weinstein introduced an alloy to which porcelain could be fused. This alloy was 84% gold and was relatively inexpensive at the time. However, in 1968, the fixed price of gold was removed by the United States Government. Wide fluctuation in the price of gold resulted and propelled the search for alternative metals into the forefront of dental materials research (Naylor, 1986).

A gold-palladium-silver alloy system was developed in 1968 in an effort to improve the high temperature strength of the gold-based alloys. Gold content was lowered, platinum was removed and silver was added. An increase in palladium content provided a greater separation of the melting range of the alloy and the fusion temperature of the porcelain. The silver content, however, was found to cause porcelain discoloration, "greening", during the firing process (Naylor, 1986).

Nickel-chromium-beryllium alloys were introduced in 1968. By 1976 they had captured 25% of the alloy market and when the price of gold reached \$800.00 per ounce in 1980 they comprised 50% of the alloy being used for dental castings (Naylor, 1986). Beryllium is added to the nickel-chromium alloys to reduce fusion temperature, improve casting characteristics, refine grain structure and add strength. Beryllium and nickel pose important health questions and although they were less expensive, the handling characteristics and physical properties of the alloys were not ideal.

In 1974, palladium-silver alloys were introduced. This system presented the handling characteristics of gold-palladium-silver alloys at a considerably lower cost. Methods to avoid porcelain "greening" were time consuming and limited the use of this alloy system (Naylor, 1986).

Gold-palladium alloys captured a significant share of the metal-ceramic market in 1977. This system was more expensive but

has excellent properties and handling characteristics (Naylor, 1986).

Because of the extreme rise in the cost of gold in 1979, the popular alternative to noble metals became nickel-chromium-beryllium alloys. Beryllium free alloys and cobalt-chromium alloys were developed in response to biocompatibility issues. Also in the 1980's, high palladium alloys were made available with 74-88% palladium and 1-2% gold. The high palladium alloys were popular among those with doubts about using base alloys, but the performance of the high palladium alternative was not up to expectations. Problems with these alloys include porcelain discoloration by oxides (graying and bluing), thermal creep with porcelain application, low sag resistance, reduced polishability, and difficult pre-soldering (Naylor, 1986).

In today's market, 50% of the metal-ceramic restorations are made with noble metal and 50% with base metals. High palladium alloys currently comprise approximately 60% of the noble alloy market. Twenty to 33 % of this group are alloys containing silver (Goodacre, 1989). Goodacre (1989) stated, "the physical properties, handling characteristics, and porcelain bonding of palladium-silver alloys have been judged to be comparable or superior to other noble metal ceramic alloys."

With base metal alloys comprising 50% of the metal-ceramic market, health questions became the focus of attention. Moffa, et.al.(1973) outlined the potential hazard of beryllium. The major health risk associated with beryllium regards an industrial



and laboratory concern. There are both acute and chronic forms of beryllium toxicity documented, and precautions are necessary for dental laboratory technicians.

The American Dental Association's Council on Dental Materials, Instruments, and Equipment reported on the potential hazards of nickel in 1982. The report outlined the problem of nickel allergic hypersensitivity and concluded "The question remains unanswered as to whether there is a danger that long-term intraoral exposure to a nickel-containing dental alloy can result in an induced nickel sensitivity." (ADA, Council on Dental Materials, 1982). The ADA Council on Dental Materials, Instruments, and Equipment again reported on base metal alloys in 1985. Although Moffa, et.al. (1983) reported no correlation between the presence of intraoral nickel alloy restorations and the incidence of nickel sensitivity, the ADA Council on Dental Materials (1985) repeated the concern of beryllium toxicity and nickel allergy stating, "The systemic response to metallic nickel and nickel compounds as a result of intraoral corrosion and dissolution of nickel-based restoration over extended periods have not been studied adequately."

In a review of the literature on the health risks of base metal alloys by Pierce and Goodkind (1989), the caution is repeated, "As yet, little is known about the effects of dental alloys containing the elements of nickel, beryllium, and chromium on the human host." Zawadzki, et.al., (1989) reported on the localized effects in 460 patients with a total of 1000 units of

nickel-chromium and palladium-silver alloys. Their conclusion was: "in general the "trends" that appeared among the alloys were neither statistically nor clinically significant."

An alternative metal with proven biocompatibility is titanium (Ti). The tissue response to implants containing 90% or more Ti exhibit a very significant increase of Ti in the surrounding tissue without sequelae (Ferguson, et.al., 1960). Williams (1981) explained, "The biocompatibility of any metal is governed by the rate of corrosion and the toxicity of the metal ions.". He described the tissue containing Ti ions as ... "always viable with multi-nucleated giant cells seen only rarely." and stated, "There is no evidence to suggest that Ti can cause hypersensitivity reactions.". Kasemo (1983) reported on the oxide layer that forms to a 10 angstroms thickness in less than a millisecond and to 50 - 100 angstroms in one minute. The oxide is considered an "oxide ceramic" and is composed of  $Ti_2O_3$ ,  $TiO$ ,  $Ti_2O$ , and may be considered to be  $Ti_2O_3$ . Because of this rapid biocompatible oxide formation, the metal is said to "passivate". Hansson, et.al.(1983) described the favorable response of bone and epithelium to Ti and Albrektsson (1983) attributed the development of ordered haversian systems and hemidesmosomal attachments to the tightly adherent oxide ceramic layer. In 1985, Toth, et.al. reported, "To date, no toxic reaction to either commercially pure titanium or titanium alloy (Ti-6Al-4V) implants have been reported...There are no reports in the literature of primary tumors arising in association with any dental

implant...There have been no reports of allergic responses to pure titanium or titanium alloy.". Ravnholt (1988) conducted a study in vitro coupling Ti with gold alloy, cobalt-chromium alloy, stainless steel, a conventional amalgam, and a high copper amalgam in a simulated oral environment. Corrosion products and increased Ph were recorded with the Ti / amalgam combinations but no change was detected with the other metals. Ravnholt concluded that when galvanically coupling Ti with corrodible metallic dental materials potentially harmful effects of increased Ph and current should be considered. However, Rupp, et.al., (1986) reported an in vivo use of Ti in proximal contact with a gold crown and in occlusion with an amalgam restoration with no indication of galvanic reaction, unusual taste, or discomfort.

### C. Development of Titanium

Titanium was discovered in 1789 by W. Gregor (Hruska, 1987). Titanium is the ninth most abundant element in the earth's crust. It exists in the form of the stable minerals ilmenite (53% titanium dioxide) and rutile (100% titanium dioxide) and does not exist in the metallic state because of its affinity for oxygen and other elements. The production of elemental Ti is complicated by its reaction with oxygen, nitrogen, and moisture as well as carbon and most refractory materials. Ti sponge is produced by the Kroll process, reduction of titanium tetrachloride with magnesium metal in an inert atmosphere. This produces 99.5% pure

titanium. There are about 30 commercially available grades of pure Ti and Ti alloys. Ti-6AL-4V accounts for 50% of the total mill products used (IPCS, 1982).

World production of titanium in 1979 involved 14 countries with 86% of the global production of ilmenite in 4 countries (Australia 33%, Norway 24%, USA 17%, and USSR 12%). Australia produces 77% of the total rutile. Titaniferrous slag used to make Ti pigment is produced by Canada (61.4%) and South Africa (38.5%). A small amount of titaniferrous slag is also produced by Japan (IPCS, 1982).

The aerospace industry consumes 95% of the Ti metal used in the United States. Titanium is also used in chemical processing, printing, pigments, paints, lacquers, ceramics, food additives, drugs, cosmetics, polymerization catalysts, organic compounds, cross-linking agents and structural metals. Titanium dioxide, an effective ultraviolet sunscreen, has been used in the treatment of herpes simplex and photosensitive cheilitis and as an anti-inflammatory ointment for gingiva and oral mucosa (IPCS, 1982).

The biomedical applications of Ti were first recognized in orthopedic implantology. Bannon, et.al.(1983) stated, "The corrosion resistance, biocompatibility, strength, and ability to be incorporated by knitting bone make titanium a strong technical choice for use in surgery. In addition, the long-range availability of the metal and geopolitical stability of the nations where it is found make titanium a cost-effective implant material." Implant components and many of the parts used for

aerospace applications are machined or forged from Ti stock. The process of machining engine parts proved to be an expensive, material wasting, time consuming endeavor. Scrap loss for typical fighter jet parts can range from 64 - 85%. This creates a "buy-to-fly" ratio that is unacceptably high (Whitt and Weaver, 1984).

The technology for casting titanium was first developed for the aerospace industry (Parr, 1985, Leisner, 1986 and Perez, 1987). . Complex parts as large as 50 inches in diameter are now being produced as one-piece investment castings. Ninety percent of the castings in the aerospace industry use the Ti-6Al-4V alloy. Large Ti castings are processed with high temperature and inert pressure (HIP processed) which homogenizes the microstructure and eliminates internal gas and shrinkage voids. The parameters used are 1650 °F and a pressure of 15 KSI for 2 hours. The lost wax process used for Ti casting in the aerospace industry is virtually identical to the process used in dentistry for the fabrication of crowns and fixed partial dentures. Wax patterns are injected into molds that have been machined from aluminum. The patterns are sprued and dipped into a "ceramic slurry" which hardens around the pattern. The wax is melted out and the mold loaded into a vacuum casting furnace. Ti electrode stock is arc-melted into a water-cooled copper crucible and poured into the hot mold (Barice, 1984).

Industrial melting of Ti is accomplished in one of two ways; in an electron beam furnace or in a vacuum arc-furnace with a

consumable electrode. A water-cooled copper crucible is used which causes a "skull" of solidified metal to grow on the inner side of the crucible. The metal is melted within itself reducing contamination. The investments used for industrial castings are described by Leisner (1984) as "extremely pure, electrographite artificial carbon with a special binder." He lists three types of molding systems:

1. graphite artificial resin binders
2. thermodynamic high solid oxides/oxide binders  
(ThO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, CaO and others)
3. high melting metals/oxide binders (W, Mo)

The patterns are only face coated with these materials to reduce costs. Centrifugal casting in a vacuum is employed to reduce porosity and quickly fill the mold. The parts are then HIP processed to heal gas holes and blowholes (Leisner, 1984).

#### D. Titanium for Dental Casting

In search of a low cost biocompatible alternative to noble metals for cast dental restorations, titanium, with highly visible success in dental implantology, became a likely choice. Titanium is highly reactive at its melting temperature of 1720°C (3128 °F) and requires an inert atmosphere to control oxidation (Ida, 1982). Casting systems employing argon atmospheres and high temperature investments have made dental restorations of commercially pure Ti, as well as Ti alloys, feasible (Ida, 1980, Greener, 1986).

## 1. CASTING SYSTEMS

Two systems for Ti dental castings are currently commercially available. One is a vacuum casting machine (CASTMATIC, Iwatani and Co., LTD., Osaka Japan). The "CASTMATIC" has an upper chamber, where the metal is melted, and a lower chamber, containing the mold. Both chambers are evacuated with a vacuum pump, then the upper chamber is filled with compressed argon gas. An argon arc is generated between a tungsten electrode and the metal, melting the metal which then, due to the pressure differential, fills the mold in the lower chamber (Ida, et.al., 1982a). The casting chamber of this system is limited in size and will not accommodate large prostheses such as removable partial denture frameworks (Szurgot, et.al., 1988).

The other system commercially available is a vertical centrifugal casting machine (Ohara, Osaka, Japan). The Ohara system employs an argon arc to melt the metal, however, it does not evacuate the casting chamber nor the mold prior to casting (Ohara, 1987). A mechanically wound coil spring capable of forces up to 170 g forces the low density Ti into the mold centrifugally (Szurgot, et.al., 1988).

A third casting system, not currently available, has been developed. Waterstrat, et.al. (1985) report this casting system to be "A relatively simple and inexpensive arc-melting apparatus... permitting the casting of any metal, regardless of its melting temperature, provided a suitable mold material is available."

In November, 1989, Hamanaka, et.al., reported on a new casting machine based on the principle of the "Castmatic". Specific improvements included: the melting and casting chambers are evacuated to a higher degree ( $2.6 \times 10^{-4}$  torr ten minutes after the mold is set in the casting chamber); a heater has been placed in the casting chamber to control the temperature of the mold; interchangeable split type and tilt type copper crucibles have been developed; a device for direct suction has been placed at the bottom of the mold chamber for improved castability; the vacuum tank and compressed argon gas tank have been set to operate more efficiently; the capacity for melting alloy is increased to about 100 g; and a new control system has been developed. With the improvements made, the authors demonstrated the ability to use a conventional phosphate bonded investment. Oxidation of the titanium is reduced because Q is removed as the mold is heated in a high vacuum.

## 2. ADVANTAGES OF TI

The use of cast Ti is relatively new to the dental profession. McLean (1983) suggests that an alternative to noble metals for use in metal ceramic restorations should have the following properties:

- high yield strength
- high modulus of elasticity
- good fatigue resistance and toughness
- ease of casting
- accuracy of thin section



- ease of soldering
- burnishability
- thermal stability and sag resistance
- tarnish resistance
- biocompatibility
- thermal and oxide compatibility with porcelain

The recent research with Ti has been looking at many of these properties. The characteristics that favor prosthodontic applications of Ti are high strength with low specific gravity, low cost, biocompatibility, and corrosion resistance. It has been used for some time in orthopedic and dental implantology, but implants are fabricated by machining, not casting. The first use of Ti as a cast dental restoration was accomplished in 1978 by alloying 82% Ti, 13% Cu, and 5% Ni. This alloy had a fusion temperature of 1538 °C (2800 °F) and was cast using conventional dental casting equipment and a commercial phosphate bonded investment (Waterstrat and Rupp, 1978). However, the questionable biocompatibility of nickel (ADA, Council on Dental Materials, 1982,1985) makes its presence undesirable. In 1984, Moser, et.al., looked at alloys of Ti with Cu, Co, and Ni. Their purpose was also aimed at lowering the melting temperature to the more acceptable range of 1370 - 1480 °C (2498 - 2696 °F).

In 1980, Ida,et.al., introduced the CASTMATIC casting system and cast pure Ti, and Ti alloys including nickel, cobalt, chromium or aluminum as binary or ternary compositions. They investigated fusing temperature, tensile strength, hardness,

castability, and tarnishability. They claimed, "Crowns and bridges of titanium, which have never been used in the dental field, were able to be cast into molds formed with dental investments." With the equipment available to make dental restorations in pure Ti and Ti alloys, research on the use of this metal began.

### 3. PORCELAIN COMPATIBILITY

The compatibility of porcelain and Ti is an important factor in the acceptance of Ti as an alternative metal. The thermal expansion coefficient of pure Ti ( $9.41 \times 10^{-6}/^{\circ}\text{C}$ ) is considerably lower than the alloys currently being used. Available porcelains have coefficients of thermal expansion between  $13$  and  $14 \times 10^{-6}/^{\circ}\text{C}$ . To take advantage of the fact that porcelain is strongest under compression, it should be  $0.5$  to  $1.0 \times 10^{-6}/^{\circ}\text{C}$  less than the expansion coefficient of the metal (Craig, 1985). Togaya, et.al.(1983) investigated the bonding of three commercially available porcelains, two mixtures of commercial porcelains, and three experimental porcelains, to commercially pure Ti. The mixtures and experimental porcelains had coefficients of thermal expansion ranging from  $8.53$  to  $9.96 \times 10^{-6}/^{\circ}\text{C}$ . The newly prepared porcelains also had fusing temperatures considerably below the commercially available porcelains ranging from  $575$  to  $730^{\circ}\text{C}$ . The increase in weight of Ti as a result of oxidation is remarkably high above  $900^{\circ}\text{C}$  and only slight below  $800^{\circ}\text{C}$ . Excess oxidation greatly decreased the bond strength of the porcelains and, therefore, the lower fusing experimental

porcelains were found to match Ti both in coefficients of thermal expansion and oxide formation. The bond strengths of the experimental porcelains with Ti was 160 kg/cm<sup>2</sup> in this study.

Riesgo, et.al. (1984) demonstrated the possibility of bonding commercial porcelains to titanium alloyed with cobalt, copper, or palladium. To control the amount of oxide formation, a vacuum of 10<sup>-4</sup> torr was used to fire the opaque layer. The normal temperature ranges for the commercial porcelains were used. Using an electron microprobe, diffusion of Ti into the porcelain and tin into the Ti was evident, indicating the feasibility of bonding porcelain to various Ti alloys.

Menis, et.al.(1986) developed three experimental porcelains for bonding to Ti. The major modifications were the addition of boric oxide (B<sub>2</sub>O<sub>3</sub>) and a decrease in the amount of potassium oxide concentrations. Reducing the potassium oxide concentration decreases leucite formation and thereby decreases the thermal expansion. Thermal expansion coefficients of the experimental porcelains ranged from 5.9 to 9.0 x 10<sup>-6</sup>/°C depending on heating rate. The bond strengths of the experimental porcelains was slightly lower than that of commercially available porcelains to gold palladium alloys. This study revealed the bond failures to be the result of the oxide formation on the commercially pure Ti.

#### 4. INVESTMENTS

With the high melting temperature of Ti (1720°C / 3128°F) and its reactivity with oxygen and nitrogen at this temperature,

the conventional phosphate bonded investments yielded significant metal/investment interactions. Ida, et.al.(1982b) compared the physical properties of castings made in magnesia investment to those made in phosphate bonded investment. Their report stated the surface characteristics were better with the magnesia investment. The metal could be properly cast into magnesia investment burned out at 800°C, and the elongation and tensile strength were unchanged by casting into molds at elevated temperatures. At higher than room temperature, castings made in phosphate bonded investment show a dramatic decrease in elongation values. Additionally, the study reported less surface hardening due to oxygen absorption with the magnesia investment. Ida, et.al. (1982a) reported on the mechanical properties of Ti cast in magnesia and phosphate bonded investments and compared them to gold, nickel-chromium, and cobalt-chromium alloys. The mechanical properties of pure Ti were found to be close to those of gold alloys, types III or IV and more ductile than Ni-Cr or Co-Cr alloys. The surface was smoother and cleaner with the magnesia investment, however, the thermal expansion curve for the two investments indicates a potential problem with the accuracy of castings made in the magnesia investment. Although the thermal expansion of magnesia is nearly linear, it is considerably lower at 800°C than the phosphate bonded investment. This would mean some type of die expansion may be necessary to fabricate castings that are not undersized.

Taira, et.al.(1985a) examined a "duplex face coat procedure" involving a primary coat of yttria ( $Y_2O_3$ ) or zirconia ( $ZrO_2$ ), a secondary coat of titanium dioxide ( $TiO_2$ ) and zirconium acetate binder, with a backing of conventional phosphate bonded investment. This report stated that castings of pure Ti in the magnesia investment yielded large concentrations of internal porosity due to the density of the investment and excessive shrinkage. Successful castings were made with both face coating materials, but finning was observed with the yttria due to facecoat cracks. In a subsequent study, Taira, et.al.(1985b) found the face coat affected the internal hardness values for pure Ti. With the yttria, an internal hardness value of 170 KHN was measured, and with zirconia, 180 KHN. Water quenching increased the values in excess of 400 KHN extending 1mm into the body of the casting. Metallographically, pure Ti was observed to be mostly alpha plus a minor beta phase. Oxygen diffusion for pure Ti extended 1mm into the casting for both the facecoating materials.

Waterstrat and Giuseppetti (1985) described a zirconia ( $ZrO_2$ ) based investment mold material to be used with their new casting apparatus. In 1986, Rupp, et.al., stated this zirconia-based investment "produces nonporous, uncontaminated and thermal-shrinkage-compensated castings. The investment is raised to 1400 °C during wax burnout establishing a permanent expansion. The molten metal is then cast into a cold mold.

Greener, et.al.(1986a), looked at specimens cast in a phosphate bonded,  $Al_2O_3/SiO_2$  investment (the Ohara argon/electric arc centrifugal casting system). A comparison was made in this investigation of the commercial dental titanium alloys to the relevant ADA specifications. Ti-6Al-4V (90% Ti, 6% Aluminum, and 4% Vanadium) exceeded ADA specification #14 for removable partial dentures with respect to yield strength and elongation. Commercially pure Ti possessed properties intermediate between quenched and hardened type IV gold (ADA specification #5). By comparing the hardness values of the as-received and the as-cast specimens it was determined that there was little oxygen uptake during casting. The Ohara investment, Titanium-Vest, is a proprietary alumina-silica mixture. Szurgot, et.al.,(1988) indicate this investment is strong enough to withstand the high acceleration forces produced by the centrifuge, has expansion to match the solidification shrinkage of the titanium and is relatively unreactive with the molten titanium.

#### 5. CASTABILITY

The castability of Ti and Ti-6Al-4V was evaluated by Greener, et.al.(1986b) comparing the two available casting systems (Castmatic, Iwatani Corp., Osaka, Japan and Titaniumer, Ohara, LTD., Osaka, Japan). In this study, following manufacturers instruction, 100% castability was achieved with the Titaniumer. The Castmatic produced only 10% and 20% castability for Ti and Ti-6Al-4V respectively.

Blackman and Barghi (1989) reported on the reliability of casting Ti in a removable partial denture configuration with the Ohara Titanium. Their success rate was 90% with two defective castings out of twenty.

#### 6. MECHANICAL PROPERTIES

In an effort to improve the mechanical properties of commercially pure titanium (C.P.Ti) Luchsinger, et.al., (1985) alloyed Ti with both alpha- and beta-stabilizers. They found that a beta-stabilized alloy (70% Ti and 30% vanadium) had higher tensile strength, good elongation, and adequate hardness, but lacked castability. Taira et.al.(1986) investigated the effects of alloying titanium with certain metallic elements on mechanical properties. The alloys studied were commercially pure Ti, Ti-6Al-4V, Ti-15V, Ti-20Cu, and Ti-30Pd. According to this report, "Cast pure Ti exhibited strength values comparable to those of dental gold alloys (Table 1.). Its elongation was  $7.9 \pm 2.84\%$ ; modulus of elasticity was  $0.96 \pm 0.10 \times 10^6$  Mpa; 0.1% yield point was  $336 \pm 18$  Mpa and ultimate tensile strength was  $415 \pm 35$  Mpa." Using zirconia facecoating and phosphate bonded investment it was noted that the use of a hot mold (850°C) was necessary to compensate for metal shrinkage and avoid internal porosity due to rapid solidification. This, however, resulted in a slow oxygen diffusion which hardened the casting gradually from the surface to the interior. The Rockwell macrohardness numbers were also determined in this investigation. Cast pure Ti tested via a 1/16 inch steel ball (F scale) was  $97 \pm 1.8$  F (converted

# Mechanical Properties

TABLE 1

	UTS $\frac{\text{MN}}{\text{m}^2}$	0.2% YS $\frac{\text{MN}}{\text{m}^2}$	Elastic Mod $\frac{\text{MN}}{\text{m}^2} \times 10^3$	Elongation %	Hardness $\frac{\text{DPH, Kg}}{\text{mm}^2}$	Density $\frac{\text{gm}}{\text{cm}^3}$	Fusion Temp. $^{\circ}\text{C}$
Ti	735-883 *	383 #	102 \$	9-15 \$	190-240 *	4.51 *	1720 *
Ni-Cr	400-1000	255-730	150-210	8-20	210-380	7.5-7.7	1300-1450
Au-Pd	700-730	650-675	116-117	8-12	210-230	13.5	1270-1300
Type IV quenched	470-520	275-310	89.3	30-35	150-170	15.2	875-1000
hardened	750-790	480-510		5-7	220-250		

\* Chare Literature

\$ Togetsu, et al., 1983

# Szurgot, et al., 1988

all others Craig, 1986



Brinell Hardness Number = 112). The microstructure of the alloys tested as determined by X-ray diffraction analysis was also reported. Cast pure Ti was all alpha, ductile and soft. Cast Ti-15V was all beta, ductile and strong. The authors suggest that, "beta titanium generally has good cold-workability, burnishability, and formability, which may be more suitable than alpha titanium for future dental uses." In 1989, Taira, et.al., reported "Cast pure Ti has mechanical properties similar to those of type-4 gold alloy. Cast Ti-6Al-4V and cast Ti-15V simulated Ni-Cr and Co-Cr alloys except for the modulus of elasticity values which were 50 - 60% of those of the Cr alloys."

#### 7. ACCURACY OF CAST RESTORATIONS

The evaluation of accuracy of cast restorations has been performed by a variety of techniques. The subjective rating of crown margins by standardized evaluators has practical relevancy (Smith, 1858, Vermilyea, 1893, Nitkin, 1976, Christensen, 1966). Gustavsen (1985) used radiographs in the clinical assessment of marginal fit. These techniques, however, measure the end result of a series of casting variables (i.e. impression materials, die stone expansion, wax pattern and investment dimensional change, etc.).

A study by Sahs, et.al.(1963) and another by Pruett (1961) used mercury micromerement as an indicator of accuracy. This technique was presented to the I.A.D.R. in March 1959 by G.M. Hollenback (Pruett, 1961).

To limit the number of variables, measuring castings directly on the dies on which they were fabricated has been advocated by many investigators. Bruce (1964), Fusayama,et.al., (1966), Eden,et.al.,(1979), Vermilyea,et.al., (1983), Finger,et.al.,(1984), Hinman,et.al., (1985), Moore,et.al., (1985), and Smith,et.al., (1985) each used variable metal dies on which measurements could be made. Teteruck,et.al., (1966), Byrne,et.al.,(1986), Brukl,et.al., (1987), Davis (1988), and Schaerer,et.al. (1988) each employed stone dies on which the castings were seated, embedded, and sectioned for microscopic measurement. Brukl and Philp (1987) described "evaluating the fit of crowns on dies or tooth preparation by measuring the linear width of the cement space at various locations around the die in the sectioned plane..." as "the conventional experimental technique".

### III. METHODS AND MATERIALS

The experimental plan is based on the work of Byrne, et.al.(1986) and is consistent with the methodology used by Tetruk and Mumford (1966). Embedding castings on their respective dies and measuring the linear width of the cement space was referred to by Brukl and Philp as the "conventional experimental technique" for evaluating the fit of crowns (Brukl and Philp 1987).

#### A. Fabrication of Standard Wax Patterns

##### 1. Master die.

A metal ceramic crown preparation (Coleman 1984) was accomplished on an Ivorine maxillary canine (Columbia Dentoform Corp., New York). The preparation has a chamfer lingual margin and a 1 mm shoulder labial margin. The Ivorine tooth was prepared with diamond and carbide instruments using both high and low speed handpieces. The shoulder was finished with hand instruments and the prepared surface was polished with flour of pumice (Plate #1).

The resulting preparation has a 6 degree taper (12 degree convergence angle) as measured on a shadow graph (Figure #1). A clinical preparation was used rather than a stylized, conical preparation. This resulted in cast restorations with thick and thin areas improving the external validity of the experiment (Naylor, 1988).

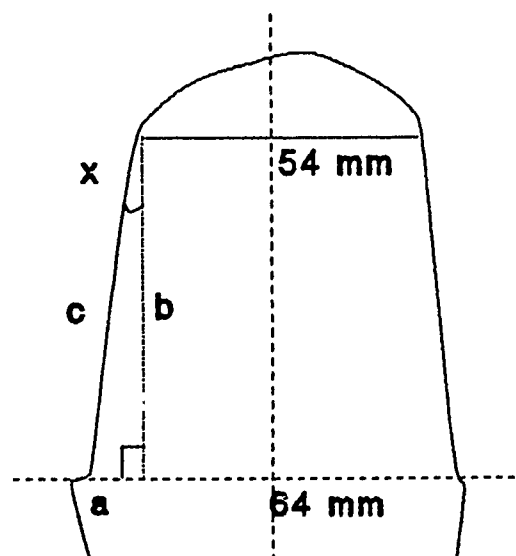
PLATE 1

PREPARATION ON IVORINE TOOTH

FIGURE 1

## SHADOW GRAPH ANALYSIS

$$\begin{aligned}A &= 5 \\B &= 51 \\C &= 51.245 \\ \sin X &= A/C \\ \sin X &= 0.0976 \\ X &= 6^\circ\end{aligned}$$



**DIE TAPER = 6 DEGREES**

A base was fabricated for the prepared tooth from plexiglass 3 mm thick and 10 mm square. The base was indexed for orientation during sectioning, and attached to the die with a screw. The root surface of the Ivorine tooth was blocked out with baseplate wax to facilitate subsequent removal from elastomeric impression material.

A stone die was fabricated by making an addition reaction silicone impression (Mirror 3 Extrude, low viscosity, Sybron/Kerr, Emeryville, Ca. 94608) in a polyvinyl chloride (PVC) tube 40 mm long with an outer diameter of 26 mm and an inner diameter of 22 mm. The PVC was painted with Kerr Mirror 3 adhesive. Silky Rock die stone (Whip Mix Corporation, 361 Farmington Ave., P.O. Box 17183 Louisville, Kentucky 40217) at a liquid : powder ratio of 22 : 100 was vacuum mixed for 15 seconds and poured into the impression. A small screw was placed in the base of the stone die before it set to facilitate the removal of the die from the impression material. This stone die was used as the master die because of the tendency for the plexiglass base on the Ivorine tooth to loosen and thereby change the relationship of the sectioning indexes when duplicating the die from multiple impressions.

## 2. Working Dies

Ten impressions were made of the stone master die in PVC tubes using Kerr Mirror 3 Extrude, low viscosity material. Each impression was poured 5 times to produce fifty type IV stone dies (Silky Rock, Whip Mix Corp., Louisville, Kentucky).

Standardization of the dies for embedding and section was accomplished by fabricating a planing device for the die bases. A sheet of plexiglass 5 mm thick by 115 mm wide and 200 mm long was slotted with a router to produce a channel 2 mm deep and 12 mm wide. A plexiglass strip 85 mm long and 20 mm wide with a hole for the die in the middle was used to stabilize the die during planing (Plate #2). Wet/dry sand paper (400 grit) was placed in the slot and the bases of the 50 dies could then be planed to a uniform 2 mm thickness, perpendicular to the long axis of the preparation.

### 3. Master Wax Pattern

A master wax pattern was made on the stone master die by waxing the crown to full contour and then cutting it back as described by Yamamoto (1985). Die-Sep lubricant (Penwalt Jelenko Dental Health Products, 99 Business Park Drive, Armonk, New York 10504) was used and the pattern waxed in green Maves Inlay Wax (Maves Co., P.O.Box 44004, Cleveland, Ohio 41440). The cutback resulted in a pattern for a metal ceramic restoration with a midfacial thickness of 0.5 mm and a midlingual thickness of 1.0 mm. A wax rib was added to the mesiofacial to improve the castability of the facial margin through the thin area. Two small drops of wax were added to indicate the location for 18 gauge open vents for casting titanium. A small hole was placed in the mesioincisal line angle to indicate the placement of a cementation vent. A 10 gauge sprue former, 10 mm long was attached to the incisal edge of the pattern and to a rubber

PLATE 2

DIE BASE PLANING DEVICE



crucible former (conical for 1-3/4" diameter casting ring). The die and wax pattern assembly was then removed from the crucible former including the wax insert connecting the sprue former to the crucible former (Plate #3).

#### 4. Split mold

Replicas of this pattern with a 10 gauge sprue former and the insert for the crucible were fabricated in a polyvinylsiloxane (Mirror 3 Extrude, Sybron/Kerr, Emeryville, Ca.) split mold with a stone matrix by a wax injection technique to insure uniformity of patterns (Dunkin, 1972, Compagni, 1984, West, 1985, Smith, 1985, Byrne, 1986, Galun, 1986). A pilot project was undertaken to develop an accurate two piece mold for injection of standardized wax patterns. Three split molds were cast in aluminum using the lost wax technique. The patterns injected were dimensionally accurate, as measured by a wax thickness gauge (Measuring Device Iwanson Hildebrand SDI Svenska-Dental Instrument AB Box 420 s-19404 Upplands Vasby Sweden). However, the external surfaces of the patterns were rough. Also, it was difficult to achieve uniform fit of the dies in the mold due to slight variations in the die bases. Therefore, the master split mold was fabricated using Kerr Mirror 3 Extrude impression material and Silky Rock die stone (Naylor 1989). A handle was attached to the die and wax pattern assembly (Plate #4). Medium body polyvinylsiloxane impression material was

PLATE 3

MASTER WAX PATTERN

PLATE 4

FABRICATION OF TWO PIECE MOLD

placed on a glass slab and light body material was injected onto 1/2 of the die/wax pattern assembly. The assembly was then seated into the medium body material completing half the impression. The material was trimmed and a stone matrix was vacuum mixed and poured to support the impression material. The stone matrix was trimmed, indexed, and boxed to pour the second half of the split mold. A separator made of Silicone Dielectric Compound (General Electric Company, Waterford, New York 12188) and chloroform was painted over the stone matrix and impression material. Light body polyvinylsiloxane was then injected to complete the impression of the die/wax pattern assembly. Retentive paper clips were placed in the impression material and a vacuum mix of die stone was poured into the boxed mold. After trimming, the mold was separated and the master die /wax pattern assembly recovered (Plate #5).

#### 5. Injection Molding

Wax patterns for casting were formed by injecting molten wax into the split mold with a lubricated stone die in place. Maves Inlay Wax was heated to 150°F in a wax pot-injector (Jewelcraft Supply Co., 431 Isom Rd. #103, San Antonio, Texas 78216). The stone dies were lubricated with Die-Sep and placed in the split mold. The two halves of the mold were secured with a rubber band and the mold inverted over the port of the wax injector. The wax was injected under pressure for 10 - 15 seconds and the crucible former insert portion of the mold completely filled by adding wax

PLATE 5

SPLIT MOLD

with a syringe. This provided a reservoir of molten wax form which the pattern could draw while cooling. The patterns were allowed to cool for 15 minutes before separating.

Margins were re-waxed under 20x magnification (MEIJI EMT widefield stereomicroscope, Meiji - Labax Co., Ltd., Tokyo, Japan) prior to removing the patterns from the dies. Ten patterns injected on 5 different dies were measured with a wax gauge to verify consistency of pattern production. All patterns measured 0.4 - 0.6 mm at midfacial and 0.75 - 1.0 mm at midlingual.

## B. Investing and Casting

### 1. Pilot Studies

Casting parameters for the fabrication of single crowns in commercially pure titanium were unproven at the initiation of this project. A pilot study to determine values for the casting variables of sprue design and powder : liquid ratios for investments was conducted. The sprue configuration and powder : liquid ratio for the investment provided by the manufacturer were used as a starting point in the study. Castings were found to be grossly undersized and several incomplete castings were made. The most difficult area to cast was the facial margin which required the metal to flow from a thick area to a thinner area. Nineteen castings were made varying the sprue design and the powder : liquid ratio until acceptable castings were produced which required minimal fitting. The sprue design involved

placing two 18 gauge open vents at specific locations and positioning the facial surface of the crown to be the trailing edge in the centrifugal casting machine (Figure #2). The powder : liquid ratio of 100 grams : 23 cc of special liquid produced castings that required only minimal fitting to achieve marginal seating.

Powder : liquid ratio and special liquid : water ratio were the variables studied for casting the Nickel - Chromium - Beryllium (Ni-Cr) and the Gold - Palladium - Indium (Au-Pd) alloys. A single investment was selected (High - Span II, Jelenko Dental Health Products, Armonk, New York) to minimize variables in the study. Three patterns were invested at a special liquid to water ratio of 4 : 1 and cast in Ni-Cr alloy (Rexillium III Jeneric/Pentron Inc., 53 North Plains Industrial Road, P.O.Box 724, Wallingford, CT. 06492). These were judged to be undersized, requiring excessive fitting. Castings in Ni-Cr using 100% special liquid required minimal fitting. The Au-PD alloy (Olympia, Jelenko Dental Health Products, Armonk, New York) fit well when 50% special liquid was used and this was in agreement with the manufacturer's recommendations.

## 2. Controlled Variables for Patterns and Investing

All wax patterns were fabricated in groups of five. This included injection of the patterns and rewaxing the margins under 20x magnification. Each pattern was placed on a crucible former (Figure #3), sprayed with a surface tension reducer (Lavon A, Jelenko Dental Health Products, Armonk, New York) and dried using

FIGURE 2

## ***SPRUED PATTERN FOR TI CASTING***

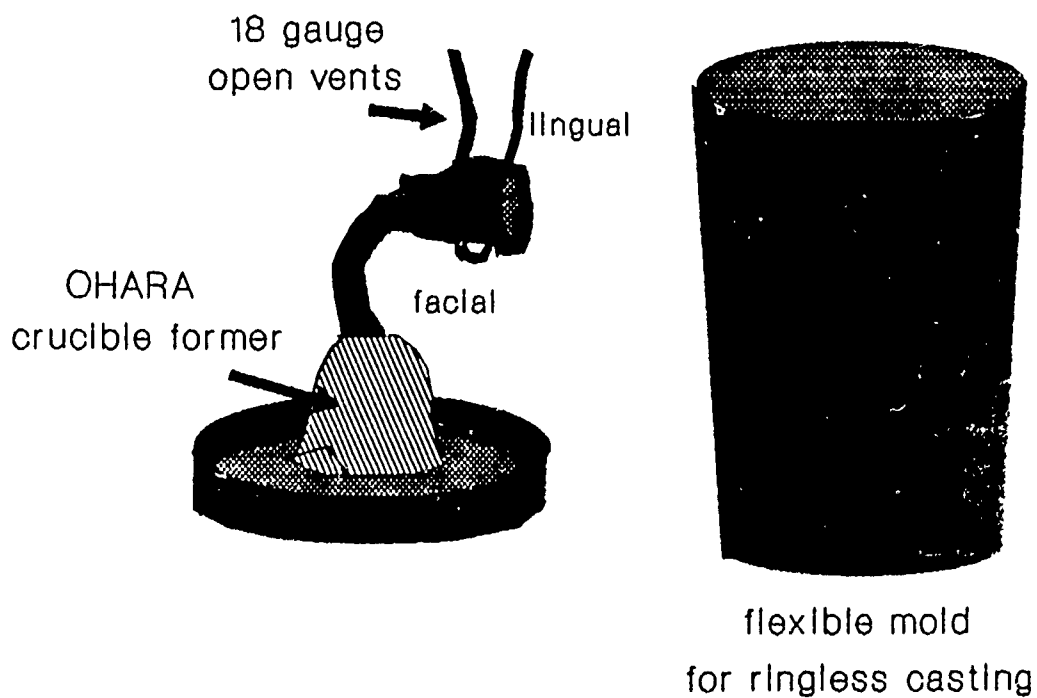
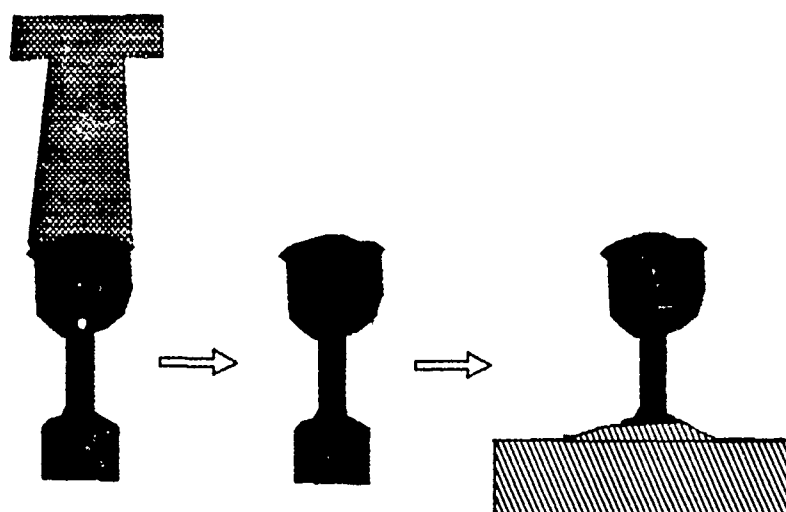




FIGURE 3

## INVESTING WAX PATTERNS



FROM DIE TO CRUCIBLE FORMER

paper points. An individual mix of investment was made for each crown, painted on the pattern with a fine brush, and vibrated into the ring. To increase setting expansion, the patterns invested for casting in titanium were immediately placed in 100 °F water and left for 30 minutes. Hygroscopic expansion was not used for the NI-Cr nor the Au-Pd castings.

### 3. Commercially Pure Titanium (Ti)

An alumina / silica based phosphate bonded investment provided by the manufacturer was used for casting the Ti crowns (Ohara, No. 5-14 Kikawanishi 3-Chome, Yodogawa-ku, Osaka, Japan). Variables including sprue and vent attachment, powder : liquid ratio, and hygroscopic expansion techniques were determined by pilot studies. A ringless casting technique was recommended by the manufacturer. The patterns were invested in plastic forms (Avtek, P.O.Box 4406, Glendale, CA 91202) which were removed prior to burnout and were reused. The Ohara crucible formers burn out with the patterns (Plate #6).

Five casting rings were processed at a time due to space limitations in the ovens. The burnout cycle for Ti castings began in a Jelenko Accu-therm oven (Jelenko Dental Health Products, Armonk, New York). The temperature was raised to 800 °C and held for 30 minutes to complete wax removal prior to moving the casting rings to the Ohara oven. This was according to manufacturer's instructions to avoid corrosion of the Ohara oven form wax residue. In the second oven the temperature was raised to 950 °C and held for 15 minutes, then raised to

PLATE 6

SPRUED PATTERN FOR TITANIUM

1200 °C and held for 15 minutes. The casting rings were then allowed to cool to 40 °C in the oven overnight with the muffle up. Castings were then made the next day into a cold mold.

Fifteen Ti crowns were cast with centrifugal force using the Ohara Titanium Casting Machine. The system uses an argon arc to melt the metal in a closed chamber flooded with argon gas. The vertical casting machine was electrically wound to 30 turns. Each 7 gram ingot of Ti was heated until it slumped and began to spark. This visual indicator of casting temperature was used to indicate the release of the casting arm.

The castings were allowed to bench cool and were divested using 25 micron aluminum oxide in a microblaster at 40 pounds per square inch pressure (psi).

#### 4. Nickel - Chromium - Beryllium alloy (Ni-Cr)

High-Span II (Jelenko Dental Health Products, Armonk, New York) was used to invest the wax patterns for the Ni-Cr alloy. The powder : liquid ratio of 90 grams of powder to 15.5 cc liquid utilizing 100% special liquid was determined in the pilot study for casting the Ni-Cr alloy. The patterns were invested as described above and allowed to bench set for 1 hour.

The casting rings used were Pyronel Casting Rings no. 4180, 1-3/4" diameter and no. 4177 rubber crucible formers (Whip Mix Corp., Louisville, KY). The glazed surface was removed from the top of the casting ring to improve the escape of gasses from the mold during casting. Five castings were burned out at a time in

a Jelenko Accu-Therm 250 oven. The temperature was raised to 1700 °F and heat soaked for 1.5 hours.

The NI-Cr alloy ingots (average weight 5.240 grams) were melted with a propane / oxygen multiorifice torch (Harris Calorific Co., Cleveland, OH, model no. 50-3) in a High-heat Quartz crucible (Williams Gold 2978 Main Street, Buffalo, New York 14214). The visual indicator to release the casting arm was when the dark shadow on the slumped ingot disappeared and the pressure of the flame caused the molten metal inside the oxide skin to move (Naylor 1986). The casting were allowed to bench cool for five minutes, then quenched in cool running water (Naylor 1986). They were divested using 25 micron aluminum oxide as described above.

#### 5. Gold - Palladium - Indium alloy (Olympia, Jelenko Dental Health Products)

The patterns for casting the Au-Pd alloy were developed and treated exactly as they were for the Ni-Cr alloy. High-Span II investment was used at a powder : liquid ratio of 90 grams of powder : 8 cc special liquid : 8 cc water. This ratio was the manufacturer's recommendation and was confirmed in the pilot study. The same casting rings and crucible formers were used for the Au-Pd and Ni-Cr alloys. The investment was allowed to bench set for 1 hour and the glazed surface was removed from the end of the ring prior to burnout. The burnout temperature for the Au-Pd castings was 1450 °F and five rings were processed at a time. The heat soak time was 1.5 hours.

Two ingots (6.2 grams) of Olympia (Jelenko) were melted with a Harris torch. The visual indicator for releasing the casting arm was when the molten alloy lost the cloudy appearance and became bright. The castings were allowed to bench cool and divested using 25 micron aluminum oxide at 40 psi.

### C. Fitting and Luting of Castings

All castings were desprued at a location indicated by a notch incorporated in the sprue former prior to the fabrication of the split mold. An ultra thin separating disc (Dedeco International, Inc. Long Eddy, N.Y. 12760) was used perpendicular to the long axis of the crown.

The castings were then examined under 20x magnification for positive bubbles that would inhibit seating on their dies. The positive bubbles were removed with a 1/4 round bur. Each crown was matched with the specific die it had be waxed on. This was insured by numbering each wax pattern and each die. The die was then sprayed with Occlude (Pascal Co., Inc., 2929 N.E. Northrup Way. P.O.Box 1478, Bellevue, WA 98009) indicator to highlight areas requiring relief. Adjustments were made under 3.5x magnification with a 1/4 round bur. Criteria for the end point of fitting the crowns were 1) apparent maximum closure of the gingival margin, 2) inverting the crown and die without dislodging the crown, 3) crown dislodging with a gentle tap of an instrument to the base of the die. Maximum seating of the crowns was verified under 20x magnification.

To remove the residual Occlude, the dies were cleaned by soaking in clear slurry (saturated dihydrate solution) and wiping with cotton. The castings were cleaned with 25 micron aluminum oxide at 40 psi and steamed cleaned.

To facilitate the embedding procedure, the crowns were luted to their respective dies with zinc phosphate cement. To minimize the effects of hydrostatic pressure impeding complete seating of the crowns a vent was placed in the mesioincisal line angle of all castings (Gerson, 1956, Bassett, 1966, Cooper, et.al., 1971, Van Nortwick, et.al., 1981, Schwartz, 1986). This vent was made with a number 4 round bur in the same location on all castings as determined by a hole placed in the master wax pattern prior to the development of the split mold. A protocol was developed for cementation to insure constancy. The powder : liquid ratio was controlled by measuring the powder on an Ohaus electronic E 120 scale with a readability to 0.001 grams (Ohaus Scale Corp., Florham Park, NJ). The ratio used was 0.8 grams : 20 drops of liquid. Cement properties of acidity, compressive strength, solubility, and retention were of no concern in this study. Therefore, the diluted powder : liquid ratio used provided a cement mix of low viscosity and minimal cement film thickness. The combination of greatly reduced cement viscosity and the occlusal venting permitted maximal seating of the crowns. All the crowns were luted to their dies during one laboratory session thereby controlling the variables of humidity and temperature. In addition, all mixes were made and all units seated by the same

operator with the same technique. The units were pushed and vibrated to place by hand, then held under a constant load of 390 grams for 15 minutes with a constant load apparatus (Plate #7).

D. Embedding and Sectioning

Polybutyrate tubing (Plastic Supply of San Antonio, Inc., 102 Josephine St., San Antonio, TX 78212) with a 3/4" internal diameter was used as a matrix to embed the die/casting assemblies. The dies were luted to a glass slab with sticky wax (Jelenko Dental Health Products) and a 1" length of the polybutyrate tubing placed over the assembly. The tubing was sealed to the glass slab with sticky wax. Ten ounces of casting resin (Castin' Craft Clear Liquid Plastic Resin, ETI, Fields Landing, CA 95537) were catalyzed with 50 drops of hardener and 150 drops of surface curing agent. The resin was mixed for 30 seconds and poured into the polybutyrate matrices to cover the die/casting assemblies. The resin was allowed to bench set for 24 hours.

The samples were sectioned using an Isomet Plus Precision Saw with an Isocut Wafering Blade #11-4265, 5"x 0.015", for iron base, cobalt base, and nickel base alloys (Buehler Ltd. 41 Waukegan Road, Lake Bluff, IL 60044). The saw was programmed for a load of 960 grams and 2000 rpm. Isocut fluid (Buehler Ltd.) was used as a lubricant during sectioning.

Each sample was cut in half faciolingually and the distal half was then sectioned mesiodistally (Figures #4 and #5). The cuts were made precisely along the indexes reproduced in the base



PLATE 7

CONSTANT LOAD APPARATUS

FIGURE 4

## MESIAL HALF SECTION

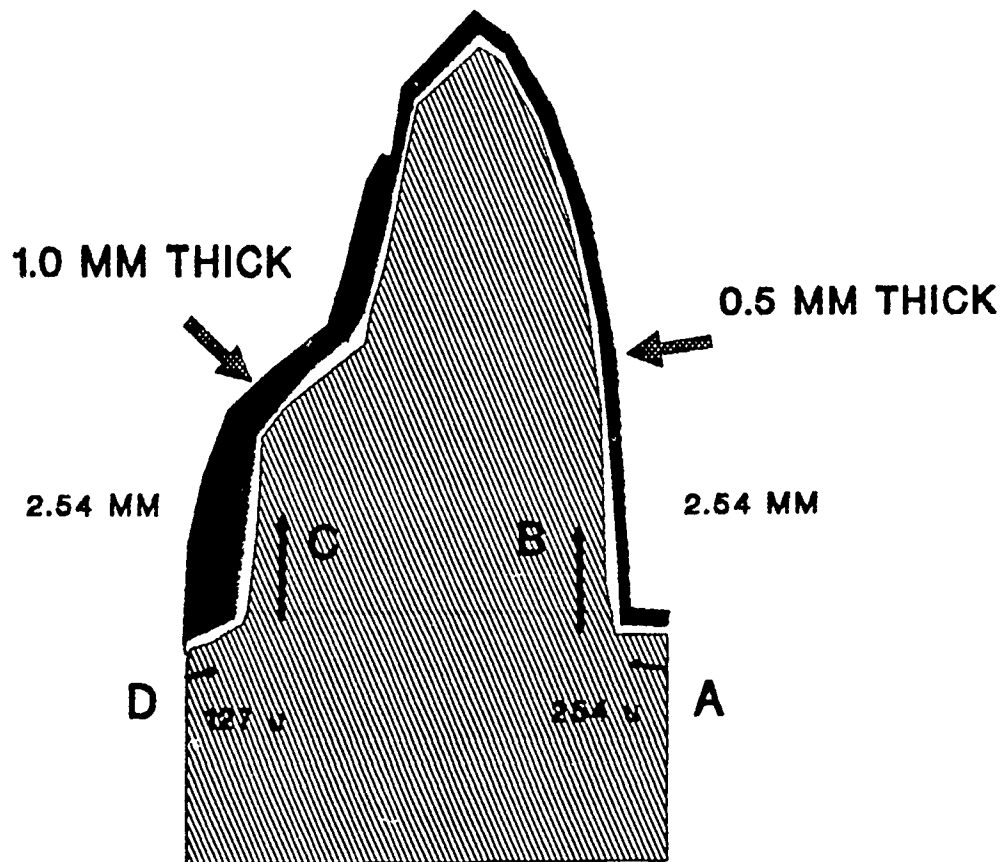
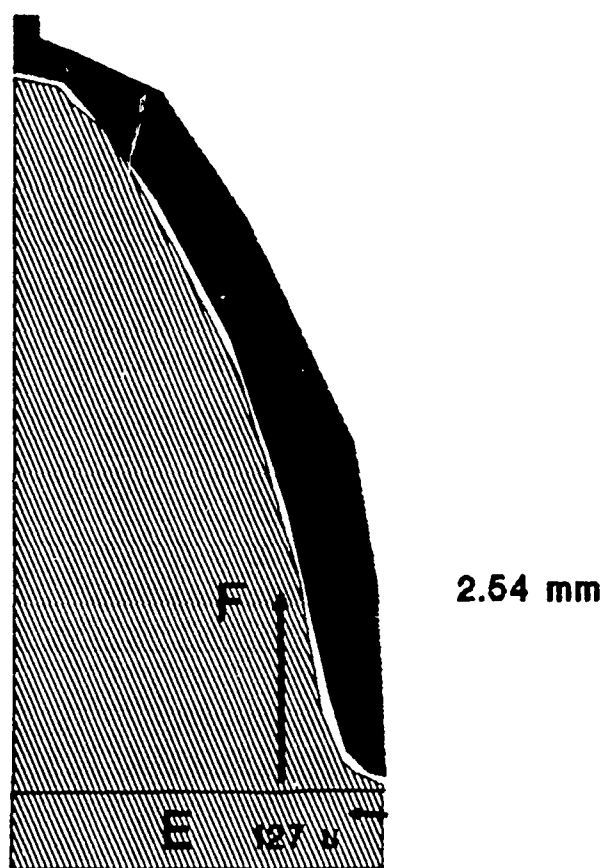


FIGURE 5

## DISTAL QUARTER SECTION



of each die to insure consistent sections. The sectioned samples were then metallographically polished through 0.05 micrometer particle Buehler Gamma Micro Polish alumina (Buehler Ltd.) on a microsoft cloth wheel.

#### E. Measuring

The linear cement space was measured on all samples with a Unitron Universal Measuring Microscope (Unitron Instruments, Inc. 175 Express Street, Plainview, N.Y. 11803). Four specific locations were measured on the mesial half section and two locations on the distal quarter section (Gravelis, 1981, Byrne, 1986). The locations are labeled A through F on Figures #4 and #5 and were determined to be as follows:

A = 254 micrometers ( $100 \times 10^{-4}$  inches) in from the facial margin

B = 2.54 millimeters (0.1 inch) up from the facial margin

C = 2.54 millimeters up from the lingual margin

D = 127 micrometers ( $50 \times 10^{-4}$  inches) in from the lingual margin

E = 127 micrometers in from the distal margin

F = 2.54 millimeters up from the distal margin

Each measurement was made three times and averaged.

The accuracy of a cast restoration is clinically determined by its adaptation at the margins. Christensen (1966) reported "Gingival margins with openings up to 119 micrometers were accepted while proximal or occlusal margins with as little as 26 micrometer openings were rejected." These results were

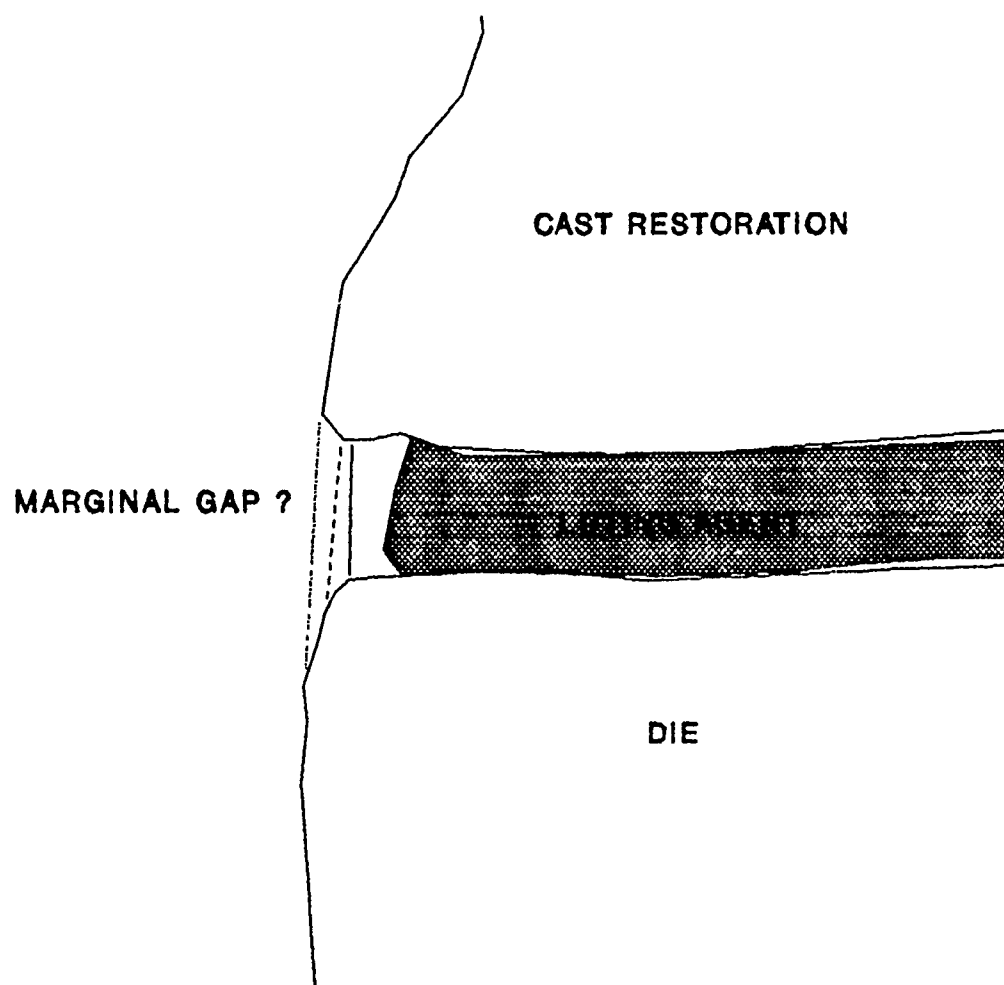
determined by the subjective evaluation of ten experienced dentists using a sharp explorer and radiographs. In the laboratory marginal openings are difficult to determine due to the "rounding" of the cavosurface of the preparation as well as the cast margin. It is difficult to determine a consistent location to be measured on the circumference of the rounded margin from sample to sample (Blackman, 1990, Figure #6). The locations chosen for measurement in this study, therefore, were selected to be representative of the potential marginal openings (points A,D, and E) and the axial adaptation of the castings (points B,C, and F) (Figures #4 and #5). Each point could be consistently determined from sample to sample on the measuring microscope by placing the cross hairs on the ocular tangent to the die and casting margins as a reference point.

#### F. Statistical Analysis

The data was analyzed with a single factor (ANOVA) for the three metals. The Tukey - B Multiple Range Test was used for specific comparisons. Comparisons of the cement space were made for:

1. Total Space = points A,B,C,D,E, and F
2. Marginal Space = points A,D, and E
3. Axial Space = points B,C, and F
4. Space at the Shoulder Margin = point A
5. Space at the Chamfer Margin = points D and E

FIGURE 6



A two factor (ANOVA) was used to compare the shoulder versus the chamfer margins for titanium. The results of the statistical analysis are given in tables #2 through #8.

#### IV. RESULTS

There was no statistically significant difference for the three metals in the measurement of cement space axially (F probability = 0.9993). The F probability for the shoulder measurement was 0.0611, also not statistically significant. Therefore the Null Hypothesis can be accepted for these two measurements but with less certainty for the shoulder measurement. In all other measurements Rexillium III was significantly different.

The Two Factor (ANOVA) resulted in an F probability factor of 0.057 for the comparison of the shoulder and chamfer margins of the titanium samples. A greater than 0.050 F probability is statistically not significant and, thereby, the Null Hypothesis is accepted. However, inspection of the data reveals a standard deviation of 5.492 for the chamfer measurement and 13.102 for the shoulder measurement. This indicates a greater variance in the shoulder measurement taken at one location (point A) which was potentially partially negated by the more stable chamfer measurement derived from two locations (points D and E).

The resulting mean cement space for Ti, as shown graphically in Figure 7, fell between the values for the Au-Pd and Ni-Cr alloys. Statistically, the values for Ti were not significantly different when compared to the "gold standard" Au-Pd alloy.

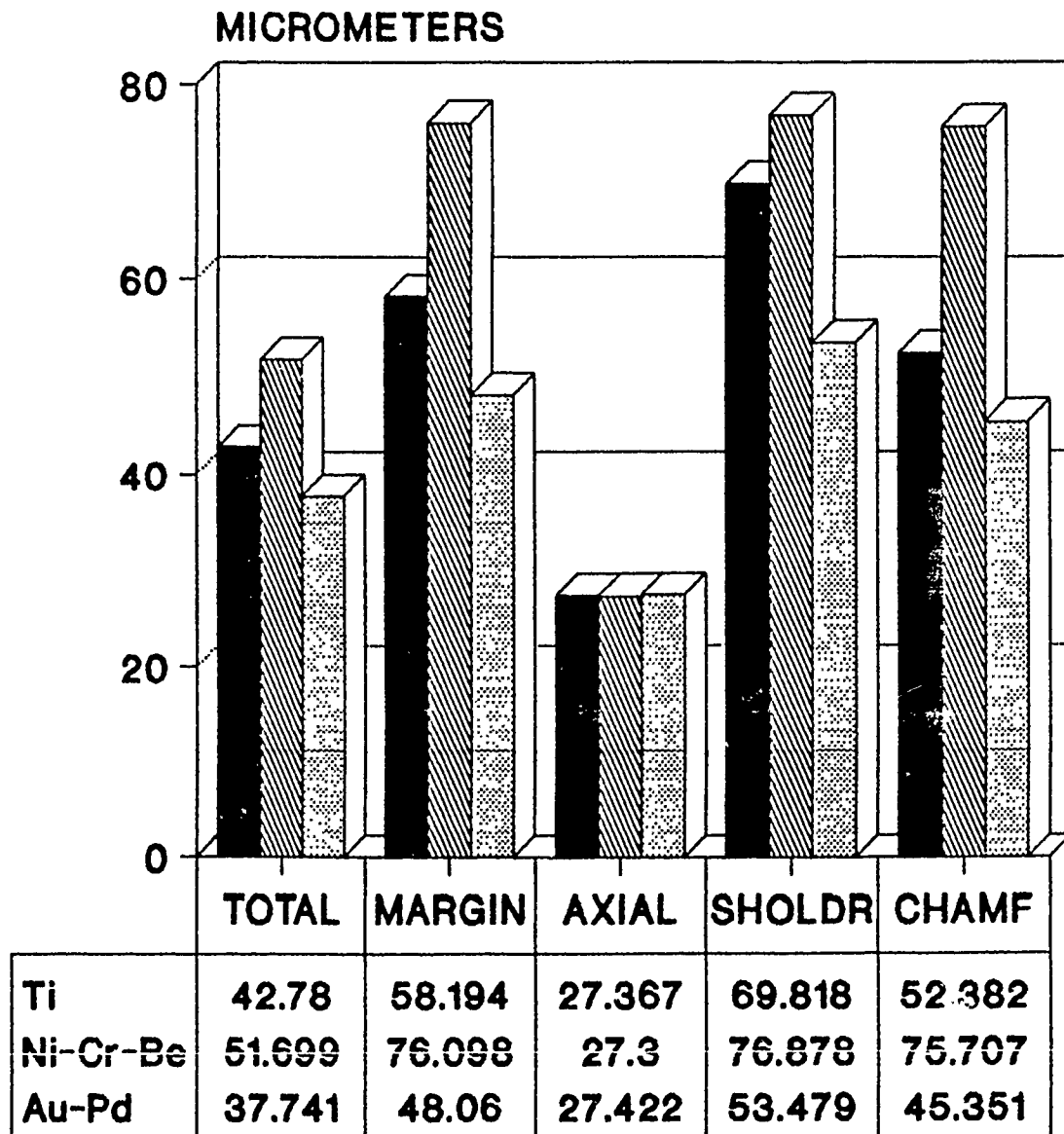


TABLE 2

RESULTS ANALYSIS (mean cement space in $\mu\text{m}$ )			
	Ti	Ni-Cr-Be	Au-Pd
TOTAL SPACE [A+B+C+D+E+F/6]	42.78	51.699	37.741
MARGINAL SPACE [A+D+E/3]	58.194	76.098	48.06
AXIAL SPACE [B+C+F/3]	27.367	27.30	27.422
SHOULDER SPACE [A]	69.818	76.878	53.479
CHAMFER SPACE [D+E/2]	52.382	75.707	45.351

FIGURE 7

## ACCURACY OF CASTING MEAN CEMENT SPACE



Ti
  Ni-Cr-Be
  Au-Pd

TABLE 3  
SINGLE FACTOR ANALYSIS OF VARIANCE  
(for three metals)

MEAN TOTAL SPACE

GROUP	COUNT	MEAN 10 <sup>-4</sup> (μm)	STANDARD DEVIATION 10 <sup>-4</sup> (μm)	STANDARD ERROR
TITANIUM	15	16.8426 (42.7803)	2.9933 (7.6029)	0.7729
REXILLIUM III	15	20.3538 (51.6987)	4.2164 (10.7097)	1.0887
OLYMPIA	15	14.8588 (37.7414)	4.5161 (11.4709)	1.1661
TOTAL	45	17.3517 (44.0734)	4.5029 (11.4374)	0.6713

SOURCE	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob
BETWEEN GROUPS	2	232.2954	116.1477	7.3927	.0018
WITHIN GROUPS	42	659.8697	15.7112		
TOTAL	44	892.1651			

MULTIPLE RANGE TEST  
(Tukey - B)

\* denotes pairs of groups significantly different at the 0.050 level

14.8588	Olympia
16.8426	Titanium
** 20.3538	Rexillium III

(ranges for the 0.050 level 3.15 and 3.43)

TABLE 4  
SINGLE FACTOR ANALYSIS OF VARIANCE  
(for three metals)

MEAN MARGINAL SPACE

GROUP	COUNT	MEAN 10 <sup>-4</sup> " (μm)	STANDARD DEVIATION 10 <sup>-4</sup> " (μm)	STANDARD ERROR
TITANIUM	15	22.9109 (58.1938)	5.9962 (15.1541)	1.5405
REXILLIUM III	15	29.9596 (76.0975)	6.9116 (17.5555)	1.7846
OLYMPIA	15	18.9213 (48.0602)	6.6708 (16.9438)	1.7224
TOTAL	45	23.9306 (60.7839)	7.8731 (19.9977)	1.1736

SOURCE	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob
BETWEEN GROUPS	2	937.2130	468.6065	10.9945	.0001
WITHIN GROUPS	42	1790.1274	42.6221		
TOTAL	44	2727.3404			

MULTIPLE RANGE TEST  
(Tukey - B)

\* denotes pairs of groups significantly different at the 0.050 level

	18.9213	Olympia
	22.9109	Titanium
**	29.9596	Rexillium III

(ranges for the 0.050 level 3.15 and 3.43)

TABLE 5  
SINGLE FACTOR ANALYSIS OF VARIANCE  
(for three metals)

MEAN AXIAL SPACE

GROUP	COUNT	MEAN 10 <sup>-4</sup> " (μm)	STANDARD DEVIATION 10 <sup>-4</sup> " (μm)	STANDARD ERROR
TITANIUM	15	10.7742 (27.3665)	2.8537 (7.2484)	0.7368
REXILLIUM III	15	10.7480 (27.2999)	2.8170 (7.1552)	0.7273
OLYMPIA	15	10.7962 (27.4224)	4.5487 (11.5537)	0.1745
TOTAL	45	10.7728 (27.3632)	3.4205 (8.6881)	0.5099

SOURCE	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob
BETWEEN GROUPS	2	.0175	.0087	.0007	.9993
WITHIN GROUPS	42	514.7774	12.2566		
TOTAL	44	514.7948			

MULTIPLE RANGE TEST  
(Tukey - B)

\* denotes pairs of groups significantly different at the 0.050 level

10.7962	Olympia
10.7742	Titanium
10.7480	Rexillium III

(ranges for the 0.050 level 3.15 and 3.43)

TABLE 6  
SINGLE FACTOR ANALYSIS OF VARIANCE  
(for three metals)

MEAN SHOULDER SPACE

GROUP	COUNT	MEAN 10 <sup>-4</sup> " (μm)	STANDARD DEVIATION 10 <sup>-4</sup> " (μm)	STANDARD ERROR
TITANIUM	15	27.4873 (69.8178)	13.1022 (33.2496)	3.3830
REXILLIUM III	15	30.2667 (76.8775)	8.9527 (22.7399)	2.3116
OLYMPIA	15	21.0547 (53.4790)	9.1802 (23.3177)	2.3703
TOTAL	45	26.2696 (66.7249)	11.0527 (28.0739)	1.6476

SOURCE	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob
BETWEEN GROUPS	2	699.8242	334.9121	2.9895	.0611
WITHIN GROUUPS	42	4705.3132	112.0313		
TOTAL	44	5375.1374			

MULTIPLE RANGE TEST  
(Tukey - B)

\* denotes pairs of groups significantly different at the 0.050 level

21.0547    Olympia  
27.4873    Titanium  
30.2667    Rexillium III

(ranges for the 0.050 level 3.15 and 3.43)

TABLE 7  
SINGLE FACTOR ANALYSIS OF VARIANCE  
(for three metals)

MEAN CHAMFER SPACE

GROUP	COUNT	MEAN 10 <sup>-4</sup> " (μm)	STANDARD DEVIATION 10 <sup>-4</sup> " (μm)	STANDARD ERROR
TITANIUM	15	20.6227 (52.3817)	5.4921 (13.9499)	1.4181
REXILLIUM III	15	29.8060 (75.7074)	9.1852 (23.3304)	2.3716
OLYMPIA	15	17.8547 (45.3510)	7.3442 (18.6543)	1.8963
TOTAL	45	22.7611 (57.8133)	8.9604 (22.7595)	1.3357

SOURCE	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob
BETWEEN GROUPS	2	1174.1490	587.0745	10.4543	.0002
WITHIN GROUUPS	42	2358.5667	56.1564		
TOTAL	44	3532.7157			

MULTIPLE RANGE TEST  
(Tukey - B)

\* denotes pairs of groups significantly different at the 0.050 level

17.8547	Olympia
20.6227	Titanium
** 29.8060	Rexillium III

(ranges for the 0.050 level 3.15 and 3.43)

TABLE 8  
TWO FACTOR ANALYSIS OF VARIANCE  
(for three metals)  
SHOULDER v.s. CHAMFER SPACE

SHOULDER VARIABLE						
FACTOR	CODE	MEAN	STD.DEV	N	95% CONF.INTERVAL	
Metal	Ti	27.487	13.102	15	20.232	34.743
Metal	Ni-Cr	30.267	8.953	15	25.309	35.224
Metal	Au-Pd	21.055	9.180	15	15.971	26.138
For entire sample		26.270	11.053	45	22.949	29.590
-----						
-----						

CHAMFER VARIABLE						
FACTOR	CODE	MEAN	STD.DEV	N	95% CONF.INTERVAL	
Metal	Ti	20.623	5.492	15	17.581	26.664
Metal	Ni-Cr	29.806	9.185	15	24.719	34.893
Metal	Au-Pd	17.855	7.344	15	13.788	21.922
For entire sample		22.761	8.960	45	20.069	25.453

TESTS INVOLVING "LOCATION" WITHIN-SUBJECT EFFECT

SOURCE OF VARIATION	SS	D.F.	MS	F	SIG OF F
WITHIN CELLS	3039.05	42	72.36		
LOCATION	276.96	1	276.96	3.83	.057
METAL BY LOCATION	154.86	2	77.43	1.07	.352



## V. Discussion

Webster (1971) defines accuracy as the "degree of conformity of a measure to a standard or a true value". To a clinician, accuracy of a cast restoration equates to the feel of the margins with an explorer (Christensen, 1966). In this study, accuracy is defined as the degree of conformity of measurements of the cement space relative to the standard Au-Pd alloy.

The accuracy of cast restorations can be evaluated by several different techniques as delineated in the review of the literature (page 24-25). The technique chosen for this study enhances the external validity of the results. A clinical crown preparation and clinical coping were used to expose potential problems with casting the metal through thick and thin areas in the pattern. Most clinical situations will require cast restorations not uniform in wall thickness and geometry. Therefore, although a symmetrical thimble shaped research pattern with even wall thickness may increase the internal validity of a study by improving consistency between samples, the results will not apply to clinical situations.

To examine the effects of the casting variables without the effects of change in dimension of impression materials or die stones, the castings were seated on their respective dies. Ideally, divesting the casting, placing it on the die and measuring the space would provide a comparison of accuracy. All castings, however, must be adjusted to fit either by selective

grinding, die spacing, or over expansion. To control for this variable, a standardized technique of selective grinding was used to achieve complete seating of the crowns.

A luting agent is required to maintain the relationship of the cast restoration to the die during sectioning and measuring. The variable of hydraulic resistance was controlled for by venting. The variable of film thickness was controlled for by a mixer the viscosity of the luting agent.

at specific locations around the ed to indicate the relative mean cement space (Gravelis, 1981, Byrne, 1986, and Brukl, 1987). The axial measurements for the three groups were not significantly different. This indicates all three groups of castings were undersized and therefore, the taper of the die determined the extent of occlusal displacement (Windler, 1979). Presumably, without luting agent the casting would seat until stopped by the axial wall, which would not close the marginal gap (27 micrometers vs. 50 to 80 micrometers).

The mean cement space at the margins is the clinically significant data. The data reveals no statistically significant difference between the mean cement space of commercially pure Ti and the Au-Pd alloy. There was a significant difference between the Au-Pd and the Ni-Cr-Be alloys. High noble metal alloys are considered to be the "gold standard" for comparison in dentistry (Eden, 1979 and McLean, 1983). Base metal alloys enjoy 50% of the current commercial market of cast restorations. In this

study, commercially pure Ti was not statistically different from the "gold standard" and statistically more accurate than the base metal alternative.

In this study there was a 20% miscast rate for the Ti castings. The area miscast was the facial margin in all three cases. Although the data for the shoulder vs. the chamfer margin when compared via the two factor (ANOVA) was not statistically significant, there was more variance in the shoulder measurements. The pattern design was altered in the pilot studies to enhance the castability of the shoulder margin through the 0.5 mm thick facial wall. These findings suggest a castability problem with this system and requires more research.

All of the Ti castings when compared to the other two groups lacked surface smoothness. Ogura (1981) determined internal roughness has an adverse effect on fit of cast restorations. The surface roughness is likely the result of the metal/investment interaction of Ti at its high casting temperature. This oxidation of the Ti surface is being addressed in the development of new casting systems (Hamanaka, 1989) and improved investments (Taira, 1985a and Rupp, 1986).

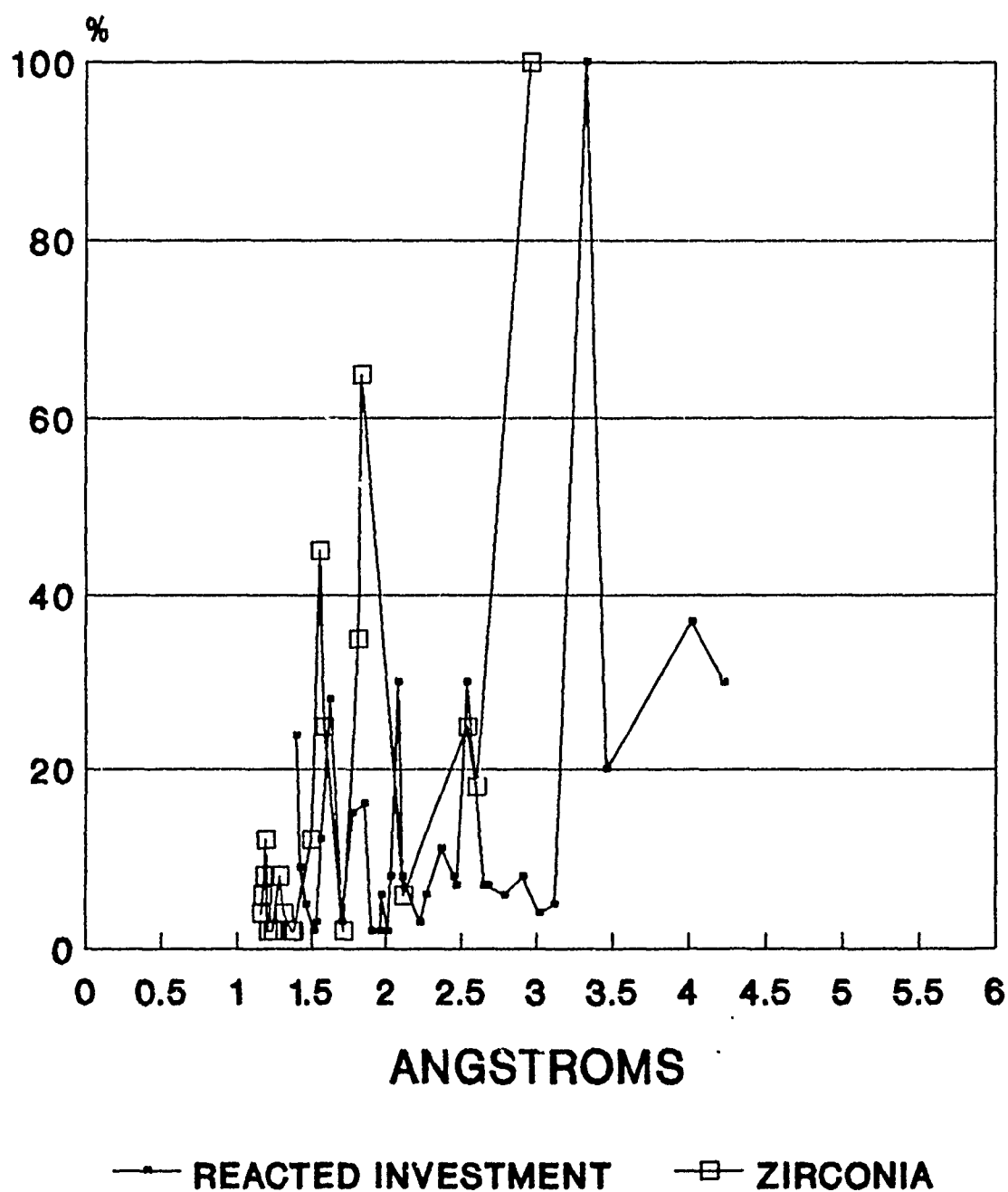
In this investigation, X-ray diffraction analysis was performed on both the unmixed components and the reacted proprietary investment, Titanium-Vest (OHARA, No.5-14 Kikawanishi 3-Chome, Yodogawa-ku, Osaka, Japan). The specific chemical composition of the investment was not discovered, however, the tracings were not consistent with either zirconia or magnesia.

Several of the peaks on the tracings were consistent with aluminum ortho phosphate, supporting the reports in the literature by Greener, et.al (1986a) and Szurgot et.al.(1988) that Titanium-Vest is a phosphate bonded, alumina/silica investment (Figures 8,9,and 10). Investment variables such as chemistry, powder : liquid ratios, mixing times and temperatures, hygroscopic and thermal expansions, and inert atmospheres, require more research.

Commercially pure Ti presents some difficult casting problems. More research is needed in castability, sprue design, investments, and casting systems.

FIGURE 8

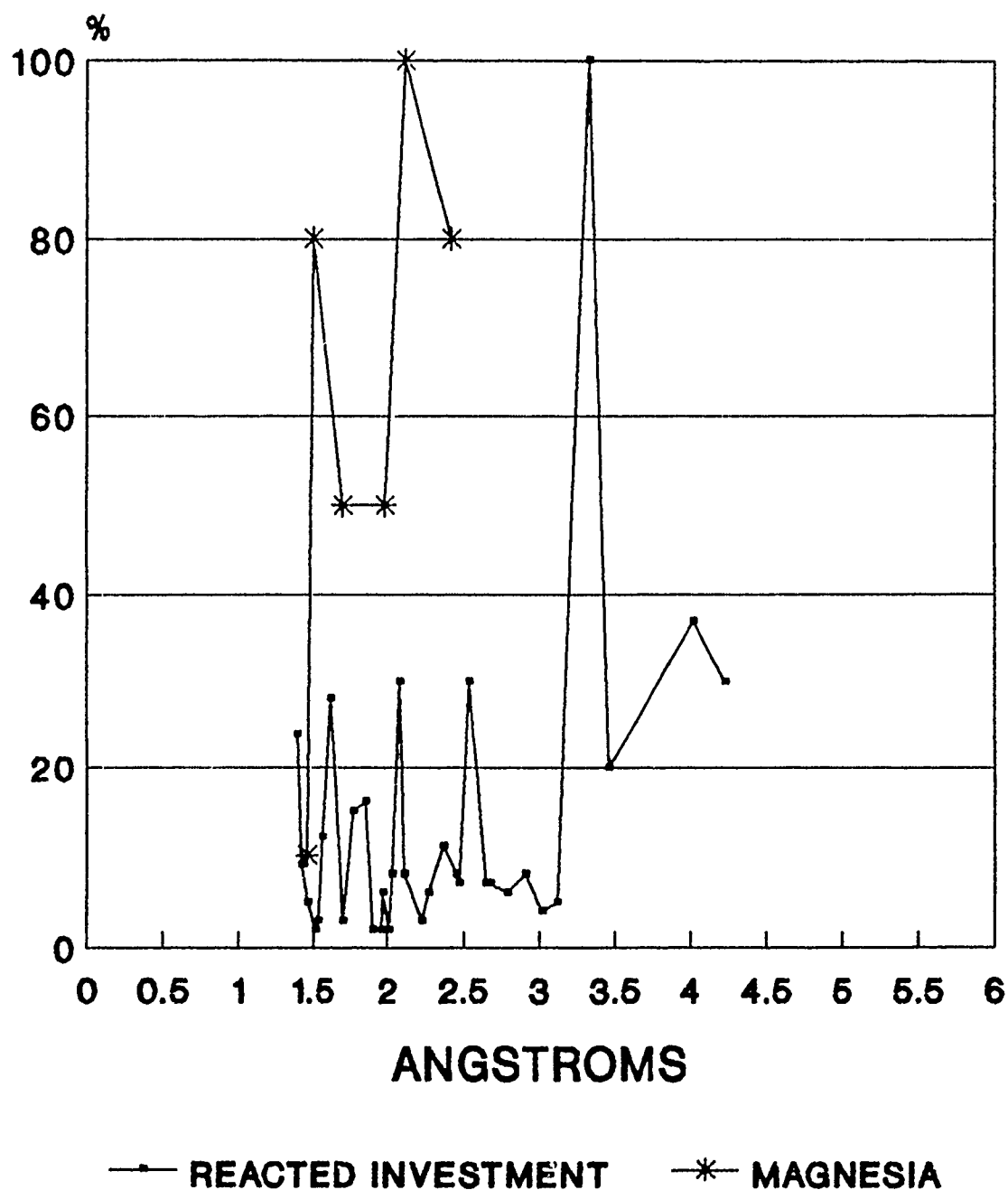
# X RAY DIFFRACTION ANALYSIS TITANIUM-VEST



(OHARA)

FIGURE 9

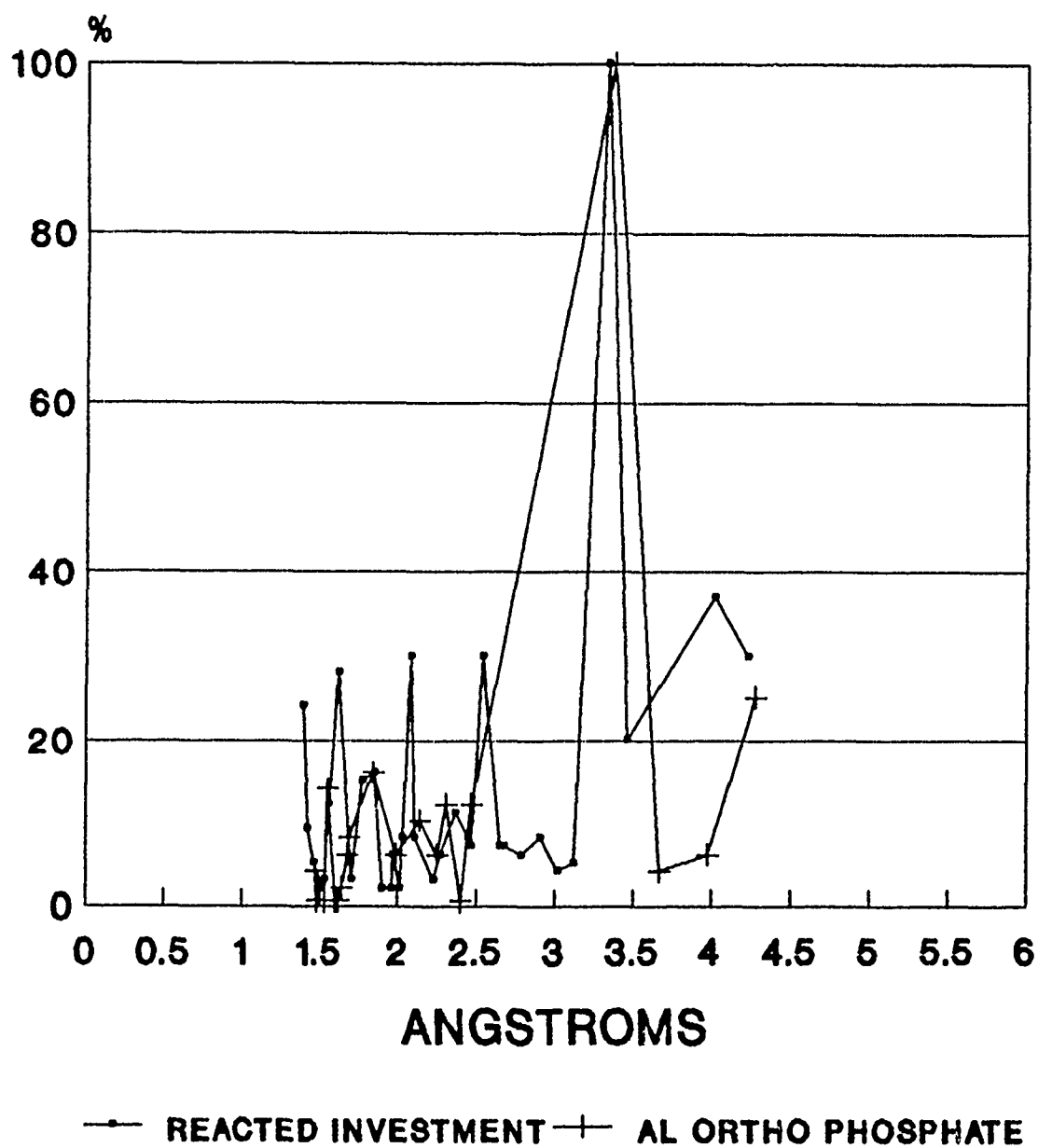
# X RAY DIFFRACTION ANALYSIS TITANIUM-VEST



(OHARA)

FIGURE 10

# X RAY DIFFRACTION ANALYSIS TITANIUM-VEST



(OHARA)

## VI. SUMMARY

The casting accuracy of commercially pure titanium (Ti) was investigated. Cement space was measured on embedded and sectioned castings with a measuring microscope. The mean cement space of Ti was compared with that of a gold-palladium alloy (Au-Pd) and a nickel-chromium alloy (Ni-Cr-Be).

The following conclusions were drawn:

1. The overall mean cement space, marginal cements space, and axial cement space for Ti was not statistically different from the Au-Pd alloy.
2. The overall cement space and marginal cement space for the Ni-Cr-Be alloy was significantly different from the Au-Pd alloy.
3. The data values for Ti fell between the data values for Au-Pd and Ni-Cr-Be alloys
4. Titanium can be considered to have a casting accuracy comparable to that of the Au-Pd alloy and better than that of the Ni-Cr-Be alloy in this study.



## APPENDICES

A. Raw Data for Titanium .....	77
B. Raw Data for Nickel-Chromium Alloy .....	79
C. Raw Data for Gold-Palladium Alloy .....	81

TABLE 9

TITANIUM X 10 <sup>11</sup>								
SAMPLE	A	B	C	D	E	F	X	μ
1	21.00	9.00	13.00	18.00	22.00	12.00		
	18.50	8.50	12.50	17.50	20.50	9.50		
	20.50	8.50	13.50	18.00	20.50	10.50		
x	20.00	8.67	13.00	17.83	21.00	10.67	15.20	38.61
2	36.50	9.00	34.00	4.00	36.00	7.50		
	38.50	8.00	39.50	4.00	40.00	7.00		
	40.00	8.50	38.00	4.50	39.50	8.00		
x	35.00	8.50	37.17	4.17	38.50	7.50	21.81	55.40
3	66.00	4.50	9.00	24.00	17.50	9.00		
	62.50	2.50	8.00	24.50	19.50	7.00		
	66.00	3.00	7.00	22.50	18.00	5.50		
x	64.83	3.33	8.00	23.67	18.33	8.50	21.11	53.62
4	21.00	7.00	10.00	28.00	18.00	17.00		
	21.00	7.50	8.50	27.00	16.00	18.00		
	20.50	7.00	8.00	30.00	14.00	18.00		
x	20.83	7.17	8.83	28.33	16.00	17.67	16.47	41.83
5	30.50	3.50	19.50	34.00	21.00	11.00		
	29.00	5.00	19.50	34.00	19.00	10.00		
	29.50	5.00	19.50	36.00	18.00	10.00		
x	29.67	4.50	19.50	34.67	19.33	10.33	19.67	49.96
6	22.00	3.50	13.50	20.00	8.50	5.50		
	23.00	2.00	18.50	19.50	9.00	6.00		
	22.00	2.50	17.50	20.50	8.00	5.50		
x	22.33	2.67	16.50	20.00	8.50	5.67	12.61	32.03
7	27.50	7.00	20.00	15.50	11.50	6.50		
	29.00	7.00	20.50	16.00	11.50	7.00		
	28.50	7.00	21.00	15.00	12.50	6.50		
x	28.33	7.00	20.50	15.50	11.83	6.67	14.97	38.02
8	13.00	13.00	13.50	21.00	12.50	11.00		
	12.50	12.50	12.50	21.00	12.00	11.50		

	13.00	11.00	12.00	21.50	12.00	11.00		
x	12.83	12.17	12.67	21.17	12.17	11.17	13.70	34.80
9	35.00	6.00	11.00	9.50	25.00	11.00		
	34.50	6.00	11.00	10.00	21.50	10.50		
	34.00	5.50	12.00	10.50	24.50	11.50		
x	34.50	5.83	11.33	10.00	24.67	11.00	16.22	41.20
10	29.00	4.50	10.00	20.50	25.50	6.50		
	29.50	4.50	10.00	20.50	25.00	6.00		
	29.50	5.50	10.00	21.00	25.00	5.50		
x	29.33	4.83	10.00	20.67	25.17	6.00	16.00	40.64
11	37.50	6.00	7.00	21.00	40.00	14.00		
	37.00	6.50	6.50	22.50	43.00	14.50		
	37.50	6.50	7.00	22.00	43.50	13.50		
x	37.33	6.33	6.83	21.83	42.17	14.00	21.42	54.41
12	26.50	6.50	9.00	10.00	11.50	13.00		
	25.00	7.00	11.00	11.00	11.50	13.50		
	26.00	7.50	10.00	10.50	12.00	12.50		
x	25.83	7.00	10.00	10.83	11.67	13.00	13.10	33.27
13	14.50	12.50	18.50	18.50	27.00	11.50		
	13.50	11.00	18.00	20.50	26.50	11.50		
	13.00	11.00	19.00	18.50	28.00	11.00		
x	13.67	11.50	18.50	19.17	27.17	11.33	16.89	42.90
14	26.00	12.00	23.50	30.50	12.50	6.00		
	26.50	12.00	23.00	29.50	12.00	6.00		
	27.00	11.50	22.50	29.00	11.50	6.50		
x	26.50	11.83	23.00	29.67	12.00	6.17	18.20	46.23
15	11.50	3.00	14.50	16.50	36.50	10.50		
	11.50	3.00	14.50	17.00	35.50	10.00		
	11.00	3.00	15.00	17.00	35.50	10.50		
x	11.33	3.00	14.67	16.83	35.83	10.33	15.33	38.94
xu	69.82	17.68	39.04	49.83	54.92	25.40		42.79

TABLE 10

REXILLIUM III x 10 <sup>-4</sup>								
SAMPLE	A	B	C	D	E	F	X	μ
16	40.50	9.50	5.00	32.00	20.00	24.00		
	41.00	9.50	6.50	31.00	19.50	24.00		
	41.00	10.50	6.00	31.50	20.00	25.00		
x	40.83	9.83	5.83	31.50	19.83	24.33	22.03	55.96
17	37.50	11.00	15.00	21.00	15.50	6.50		
	37.00	11.00	14.50	21.50	15.50	7.00		
	38.00	11.00	15.00	20.00	16.00	7.00		
x	37.50	11.00	14.83	20.83	15.67	6.83	17.78	15.16
18	24.50	6.50	15.00	29.50	44.50	6.50		
	24.00	5.50	15.00	30.00	44.50	8.00		
	24.00	7.00	16.50	30.50	45.50	7.00		
x	24.17	6.33	15.50	30.00	44.83	7.17	21.33	54.18
19	30.50	5.50	7.50	37.00	41.00	9.50		
	31.50	6.00	8.00	37.50	41.00	9.50		
	31.00	6.00	8.00	37.00	42.00	9.50		
x	31.00	5.83	7.83	37.17	41.33	9.5	22.11	56.16
20	33.50	4.50	8.50	29.00	28.50	12.00		
	30.00	5.50	7.50	29.00	25.50	11.50		
	31.50	5.50	8.00	29.50	26.00	11.50		
x	31.67	5.17	8.00	29.17	26.67	11.67	18.73	47.57
21	30.50	9.50	16.50	48.50	58.50	20.50		
	31.00	10.50	16.50	51.50	59.50	21.00		
	31.00	11.00	17.00	52.00	59.50	21.00		
x	30.83	10.33	16.67	50.67	59.17	20.83	31.42	79.81
22	28.50	5.50	12.00	23.00	15.50	7.50		
	28.50	6.00	12.50	22.00	15.00	7.00		
	29.00	5.50	12.50	23.00	15.00	7.00		
x	28.67	5.67	12.33	22.67	15.17	7.17	15.28	38.81
23	23.00	6.00	17.00	21.00	30.50	23.50		
	23.50	5.50	17.00	20.50	29.00	24.00		
	22.00	5.50	17.00	21.50	29.50	23.50		

x	22.83	5.67	17.00	21.00	29.67	23.67	19.97	50.72
24	36.50	5.50	19.50	28.50	40.50	13.50		
	38.00	5.00	20.00	29.00	40.50	13.50		
	38.50	4.50	19.00	29.50	41.50	13.50		
x	37.67	5.00	19.50	29.00	40.83	13.50	24.25	51.60
25	29.50	6.00	11.50	35.50	13.50	15.00		
	29.00	6.50	12.00	36.50	15.00	15.00		
	29.00	6.00	12.00	36.50	14.00	15.00		
x	29.17	6.17	11.83	36.17	14.17	15.00	18.75	47.63
26	29.50	4.00	12.50	15.00	32.50	7.00		
	30.00	4.50	13.00	14.00	33.00	6.00		
	30.00	4.00	13.00	14.50	32.50	6.00		
x	29.83	4.17	12.83	14.50	32.67	6.33	16.72	42.47
27	24.00	10.00	6.00	22.00	29.00	4.00		
	23.50	11.00	6.00	22.50	29.50	4.50		
	23.50	11.50	6.50	22.00	29.50	4.00		
x	23.67	10.83	6.17	22.17	29.33	4.17	16.06	40.79
28	35.50	7.50	8.00	35.55	26.00	12.00		
	35.50	8.00	7.50	35.00	26.50	12.00		
	35.50	8.00	7.50	35.50	26.50	12.00		
x	35.50	7.83	7.67	35.33	26.33	12.00	20.78	52.78
29	7.50	5.00	9.00	17.00	39.00	18.00		
	6.50	5.50	10.50	16.50	38.00	17.30		
	6.50	6.00	10.00	17.00	39.00	18.00		
x	6.83	5.50	9.83	16.83	38.67	17.67	15.89	40.36
30	43.00	8.50	11.50	37.50	25.50	19.50		
	44.00	9.00	11.00	37.50	26.00	19.00		
	44.50	8.00	11.00	37.00	25.00	18.50		
x	43.83	8.50	11.17	37.33	25.50	19.00	24.22	51.52
xp	75.89	18.25	29.97	73.56	77.88	33.68		51.70

TABLE 11

OLYMPIA x 10 <sup>-4</sup>								
SAMPLE	A	B	C	D	E	F	X	μ
31	30.50	16.00	9.50	28.00	10.50	22.50		
	29.00	17.00	10.00	28.00	11.00	21.00		
	29.50	16.50	9.50	27.00	11.00	21.50		
x	29.66	16.50	9.66	27.66	10.83	21.66	19.33	49.09
32	8.50	3.00	13.50	7.50	10.50	15.50		
	9.50	3.00	13.00	8.00	10.00	16.00		
	9.50	3.00	12.00	8.00	10.50	15.50		
x	9.17	3.00	12.83	7.83	10.33	15.66	9.00	24.89
33	30.00	10.00	10.00	5.00	21.50	8.50		
	29.50	11.50	10.50	4.50	23.00	10.00		
	29.50	11.00	10.50	4.00	21.00	8.50		
x	29.66	10.83	10.33	4.50	21.83	9.00	14.36	36.47
34	33.00	4.00	8.00	17.00	27.50	11.50		
	34.50	4.50	7.00	16.50	27.00	11.00		
	34.00	4.00	7.50	16.50	26.50	11.00		
x	33.83	4.17	7.50	16.67	27.00	11.17	16.72	42.47
35	16.50	4.00	5.00	17.50	20.00	8.00		
	15.50	4.00	5.50	17.50	19.50	8.50		
	16.50	4.50	5.00	18.00	20.00	8.00		
x	16.17	4.17	5.17	17.67	19.83	8.17	11.85	30.12
36	6.00	5.50	7.50	13.50	6.50	12.00		
	4.50	5.00	7.50	14.00	7.00	12.00		
	5.50	4.00	7.50	13.50	7.00	12.50		
x	5.33	4.83	7.50	13.67	6.83	12.17	8.39	21.31
37	13.00	3.50	10.00	20.00	15.00	11.00		
	13.00	3.50	9.50	20.50	15.00	10.50		
	12.50	3.00	9.00	21.00	14.50	10.00		
x	12.83	3.33	9.50	20.50	14.83	10.50	11.92	30.02
38	28.50	4.00	17.50	29.00	12.50	8.50		
	28.50	3.00	16.00	28.00	14.00	7.50		
	28.50	3.50	17.50	28.00	14.00	8.00		

x	28.50	3.50	17.00	28.33	13.50	8.00	16.47	41.83
39	21.50	2.00	11.50	6.50	8.00	10.50		
	21.50	2.00	11.50	5.50	7.50	11.00		
	22.00	1.50	11.00	5.50	7.00	10.50		
x	21.67	1.83	11.33	5.83	7.50	10.67	9.81	24.92
40	27.00	2.50	9.00	21.50	13.50	10.00		
	29.00	2.50	9.00	21.00	12.50	10.50		
	28.00	2.00	9.00	21.50	13.50	10.00		
x	28.00	2.33	9.00	21.33	13.17	10.17	14.00	35.56
41	13.50	10.00	14.50	16.00	29.50	14.00		
	13.00	10.50	14.00	15.50	29.50	13.00		
	13.50	8.50	14.00	15.50	29.00	14.00		
x	13.17	9.67	14.17	15.67	29.33	13.67	15.95	40.51
42	17.50	8.00	3.50	18.00	25.50	12.00		
	17.00	8.00	4.50	18.50	25.00	11.50		
	17.50	8.50	4.00	19.00	25.00	12.00		
x	17.33	8.17	4.00	18.50	25.17	11.83	14.17	35.99
43	24.50	11.50	13.00	32.50	41.00	14.00		
	24.00	12.00	12.00	31.50	41.00	13.00		
	23.50	11.50	11.50	32.50	40.50	13.50		
x	24.00	11.67	12.17	32.17	40.83	13.50	22.39	56.87
44	32.50	16.00	22.50	22.50	20.00	29.00		
	32.50	19.00	23.50	22.00	21.50	30.00		
	34.00	17.00	23.00	21.00	20.50	29.50		
x	33.00	17.33	23.00	21.83	20.67	29.50	24.22	61.52
45	14.50	15.50	15.00	16.50	5.50	16.00		
	13.00	15.00	14.50	16.00	5.50	15.00		
	13.00	15.00	14.50	16.50	5.50	16.50		
x	13.50	15.17	14.67	16.33	5.50	15.83	13.50	34.29
XII	53.47	19.74	28.42	45.47	45.24	34.11		37.72

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